

VOL. 80 No. 2042  
AUGUST 30 1958

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post 1s 9d)

# CHEMICAL AGE

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## SCIENCE AND BUSINESS

**W**HY should science and business be partners? Sir Alexander Fleck, in his address as president of the British Association for the advancement of science chose to consider business and science and the need for a proper balance between them.

Addressing the inaugural meeting of the British Association on 27 August at St. Andrew's Hall, Glasgow, Sir Alexander said he was now 'a sort of common man of science' who, lacking the detailed knowledge of the specialist, had at least an understanding of the framework within which scientific thought evolved. However, he felt that the risk of being neither pre-eminently scientist nor pre-eminently businessman might be counter-balanced by advantages that come from a non-specialist approach to science and business.

It was sad to think, he said, that in a population which depended so much on science, few people clearly understood what it was all about. The pure scientists were those who were extending the broad highway of organised knowledge, and much of our progress derived from them. Those utilising what had already been built and perhaps pausing to strengthen it here or widen it there, were the technologists or applied scientists. A few combined both functions. There was nevertheless a joint responsibility to see that the highway was properly and extensively used by all mankind. The British Association, said Sir Alexander, was now devoting itself to the additional task of securing the greater use of this highway by all types of people in these islands, particularly as we entered the so-called 'Second Industrial Revolution'. In this revolution, there was the change-over from the external properties of carbon atoms to the internal properties of the lightest and heaviest atoms in nature as a source of energy.

A second feature of this revolution was the growing interdependence of the sciences as they advanced on a broad front. It has been the prior developments in several different sciences and technologies, such as chemical rocket fuels, refractory combustion chambers and radio transmitters that have produced achievements such as the launching of earth satellites.

The creative possibilities of thermonuclear fusion energy could be regarded as symbols of man's reach into a future of boundless energy, and his grasp of such a prize could well be his greatest material achievement in the history of the world.

Scientific effort on this scale costs money, however, and Sir Alexander said that the total annual research expenditure in this country alone was estimated to be £300 million. He pointed out that such effort also costs scientists. Public expenditure on education (including universities) was now some £740 million a year. For UK universities to grow large enough to accommodate everyone who could benefit by such an education, instead of the present 3½ per cent who at present enjoy it, some £1,200 million a year would be required. These large sums, if we as a nation were to stay solvent, could only come from one source—the wealth which we produce.

Our capacity to extend our science and education, Sir Alexander rightly pointed out, largely depended on our ability to run businesses successfully. He had chosen, he said, to speak of business because business was a more comprehensive term than industry.

What characteristics should a business have if it were to survive and function effectively over a period of years? Sir Alexander suggests three. First, it must contribute to the well-being of the community and its social evolution. This it can do by producing goods or services at fair prices; by giving fair treatment in the way of remuneration and working conditions; and by giving a fair return to shareholders, so that they may be encouraged to provide for further ventures. Second, business must have the capacity to change its methods and if necessary even its objectives, thus constant alertness with regard to its products or services; anticipation of the public's needs; and ability to withstand competition. Lastly, if it was to be self-perpetuating through growth or modernisation, a business must make a profit. Only from the excess of income over expenditure could business and science be financed.

Production of wealth required investment of wealth, reminded Sir Alexander. Part came from retained profits and part from banks, but the largest volume came from the investing public. Since 1918 the US has invested half as much again in relation to its resources, as has this country, and its national product has risen at an average rate of about 3 per cent a year, against only 2 per cent in this country. During the last 10 years the amount the UK had invested in its productive source has shown an encouraging growth, resulting in the output of manufacturing industry, as a whole, increasing by about  $3\frac{1}{2}$  per cent a year since 1948, compared with the  $2\frac{1}{2}$  per cent between the wars. 'If science has laid the foundation for much of this expansion, business has provided the will and the means to complete the structure and both must continue to do so,' stated Sir Alexander.

It will be recalled that last year Professor Blackett, in his presidential address to the British Association called for technological aid to the under-developed countries. Sir Alexander thinks in terms of expediency rather than moral compulsion, and believes we must concentrate for our own survival on the development of new products and processes. With the changing export pattern and other countries now manufacturing instead of producing only food or primary raw materials, the UK is obliged to rely either on selling things no one else has learnt to make, or make as cheaply, or on marketing our skill and know-how, states Sir Alexander. Lack of proper scientific direction at various times has caused traditional industries which the UK pioneered to suffer, as for instance the organic chemicals industry.

In considering the achievement of a balanced partnership, Sir Alexander dealt with the place of the scientist in the business structure. There were six or seven separate functions: finance, commerce, personnel, operation or production, design and construction, research, etc.

It is of interest to note that Sir Alexander considers good technical people are not wasted in such jobs as plant management, and he does not agree that the employment of technical men in commercial departments is necessarily wasteful for it 'gives flexibility within the business' and can ensure smoother co-operation between sales, works and research. As training for higher administrative posts, commercial experience was invaluable to the scientist. He hoped that finance and technology would become more and more interdependent. With scope for the scientist to infiltrate throughout the whole business structure, Sir Alexander felt it was pertinent to ask, if scientists were to play an important part in business administration, was the balance between science and business likely to be upset?

As British industry is at present constituted, the balance is heavily tilted the other way. There are, in fact, still many enterprises where qualified technical men are not employed even in tasks where they could be expected to be employed, and conversely no industry was yet harmfully dominated by scientists. In part, this state of affairs is

believed to be due to the shortage of scientists and technologists and partly by historical circumstances.

The second point was that in businesses where scientists played an important part in administration, non-technical men, with a good understanding of scientific principles, were with advantage put into semi-technical jobs, hence helping to maintain a balanced partnership.

Third and most important, the balance sought had to come in great measure from within the individual himself, a balance provided by the education which the scientist and non-scientist ought to receive.

This, then, is the theme of Sir Alexander's British Association address: That the advancement of science can only be achieved with the resources which business can provide, and if business is to provide them, science must come to its aid. The truly balanced partnership, in Sir Alexander's view, would manifest itself in the growing use of science and its methods in business, and this rests with a balanced education for scientists and business men alike.

Sir Alexander's standing as an industrialist and a leader of the chemical industry, coupled with the fact that his is a large and successful organisation, call for a careful consideration of his views.

## MICROCHEMISTRY SYMPOSIUM

AS this issue of CHEMICAL AGE goes to press, 27 August the highly successful International Symposium on Microchemistry which has been held at Birmingham University since 20 August is drawing to a close. This Symposium attended by over 400 delegates, of whom a quarter were from 24 overseas countries, has resulted from the international meeting of analysts arranged in 1954.

Two years ago the Midlands Section and the Microchemistry Group jointly set up an executive committee directing and co-ordinating eight sub-committees whose task it has been to organise some 60 lectures and discussions (totalling over 45 hours), the presentation of a series of most interesting demonstrations and techniques and a fine trade exhibition.

With the rapid developments during the last few years in microchemistry to which branch the analytical school of Birmingham University has and is continuing to contribute so greatly, the emphasis has been on microchemical techniques and analyses. The founding of microchemistry and its progress in South and North America, Europe and Great Britain was ably represented in the choice of the four Plenary Lectures given by Professors F. Feigl, H. Lieb, A. A. Benedetti-Pichler and Dr. R. Belcher. The lectures, with valuable extended discussions, ranged over a wide field and were delivered by chemists whose reputation is international.

The more recent branch of radiochemistry was included; a notable section of the proceedings was devoted to emission and absorption spectroscopy, polarography, chromatography and the highly specialised but important field of biochemical methods. Chemical organic elemental and group analysis and gravimetric and titrimetric methods was not omitted, and the teaching of microchemistry was not forgotten. Indeed, at Birmingham it could not be forgotten for Dr. Belcher who joined the University 10 years ago has been responsible, with the support of Professor Maurice Stacey, for the growth of the analytical research school at Birmingham so that it is now one of the largest, if not the largest, of its kind in the world. Under Dr. Belcher's leadership it is pioneering an ambitious programme of research into sub-micro methods of organic analysis, many of which have already proved to be successful.

This issue of CHEMICAL AGE contains reports of some of the papers delivered and further reports will be appearing in next week's issue. The proceedings of the symposium containing all the papers in full and discussions will be printed in due course by Pergamon Press in one volume of approximately 450 pages.

# Significance of the Transuranic Elements to the Chemist

## 'Most Significant Advance in History of Science'

**D**ISCOVERY of nuclear fission and the transuranic elements was without question the most significant advance made throughout the history of science. This was stated by Professor H. J. Emeléus in his presidential address to the Chemistry Section of the British Association for the Advancement of Science, during the annual meeting at Glasgow (27 August to 3 September). The pattern was already beginning to emerge in current programmes for the production of nuclear power and in the ready availability of radioactive isotopes.

The chemist had played an important part in this progress. Of the future significance of the transuranic elements for the chemist, Professor Emeléus said that it appeared inevitable that a new field of chemical technology would arise in connection with power production and the processing of nuclear fuel elements.

He suggested that basic training in the special techniques of radioactivity would certainly become much more general. More specialised training would have to be given in specially equipped institutes or within the atomic energy industry itself. This field abounded in opportunity and as so many of our atom power stations were run for the most part by young men who have been in it from the beginning, there was little need to fear that scientific challenge of this work, as well as the opportunity it offered, would fail to attract graduates of the future.

### Crowning Achievement of 60 Years Work

Actual isolation of the transuranic element was the crowning achievement of a series of discoveries spread over about 60 years, and beginning with the discovery of X-rays in 1895 by Becquerel. Pierre and Madame Curie in 1898 had concluded that the penetrating rays from uranium were a new atomic phenomenon. This was soon followed by the establishment of the radioactivity of thorium, the isolation of polonium and radium and in the elucidation of the nature of the rays from radioactive substances in 1903 by Rutherford and Soddy.

In the next 20 years the nucleus of the atom and its planetary electrons had become established and radioactivity was confirmed as a nuclear property.

With the study of atomic nuclei a series of advances had culminated in the discovery of fission and the transuranic elements.

After the discovery of the neutron in 1931 it was found that bombardment of the nuclei of lighter elements with  $\alpha$ -particles could produce new nuclei which were radioactive. This had proved of special significance for, since the neutron was uncharged, it was able to approach the nucleus without encountering the repulsive forces which operate

This first Chemical Age report on the annual meeting of the British Association being held in Glasgow from 27 August to 3 September, summarises the presidential address given by Professor H. J. Emeléus, C.B.E., F.R.S. to the Chemistry Section on 28 August. Professor Emeléus is Professor of Inorganic Chemistry at Cambridge.

for a proton or  $\alpha$ -particle. The importance of this process, first used by Fermi, is, of course, that the product produced has an atomic number greater than the element taken initially. Experiments with uranium had shown that  $U^{239}$  underwent two successive  $\beta$ -decays forming in turn neptunium and plutonium. These were the first of the series of 10 transuranic elements.

The chief ways of producing the isotopes of the 10 transuranic elements were: bombardment with particles accelerated in the cyclotron or similar high-voltage machine; neutron bombardment; multiple neutron capture and bombardment with heavy ions.

The first method had been widely used and a good deal of chemical manipulation was involved in separating the product from target and from fission products which often formed simultaneously. Multiple neutron capture was possible only with high neutron fluxes, such as were available in some reactors.

Use of heavy ions was a comparatively recent development. The cyclotron or a similar machine was used. Reactions such as  $U^{238}_{92}(N^{14}, 6n)Es^{246}_{99}$  had become possible and the reaction  $Cm^{244}(C^{13}, 4n)No^{253}$  is believed to have been used in the synthesis of Element 102.

Some main points in the chemistry of the transuranic elements were considered by Professor Emeléus. Chief of these from the point of view of inorganic chemistry was undoubtedly the place which these elements were to occupy in the periodic table. First, they could form a new group of transition elements fitting below the third group (Hf-Au). The second possibility was that, from the point of view of electron

structure, they were analogous to the rare earths (Elements 57-71). For the transuranic elements it would be the 5f electron shell which was completed and this was, in fact, what happened.

Actinium was believed to have no 5f electrons. Spectroscopic evidence, which was difficult to interpret in detail for heavy elements showed that there were electrons in the 5f shell for uranium and possibly also for thorium. Arium was also shown to have these electrons. The series was, therefore, thought to begin at actinium and hence the family was called the actinides, by analogy with the lanthanides. The probable number of f electrons for the two series was indicated by Professor Emeléus.

It might be expected that the actinide elements resemble the lanthanides chemically in forming trivalent ions by the loss of a single 6d electron and the pair of 7s electrons in the two outermost shells. The analogy broke down here, however. Oxidation states known at present were shown. During the exploration of this field, many new techniques had been developed. It had involved work with radioactive species of all sorts. Two special techniques, which had contributed greatly to the study of the whole field of transuranic element chemistry were solvent extraction and the use of ion-exchange resins. Solvent extraction was the basic operation in preparing pure uranium compounds for atomic energy purposes.

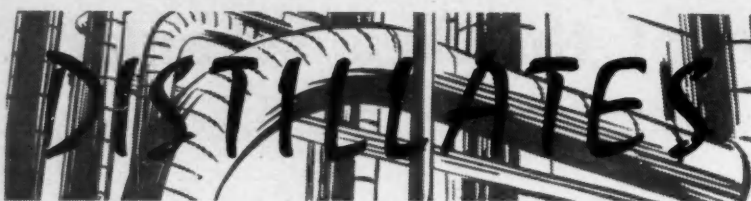
### Separating the Heavier Transuranic Elements

Use of cation-exchange resins was one of the best ways of separating the rare earth ions and the same method had been used to separate the heavier transuranic elements from one another and from rare earth fission products with which they were often associated.

The chemistry of the elements beyond curium had been established entirely by tracer techniques and the professor suggested that it was probable that in time, work on a macroscopic scale for solid compounds and elements up to einsteinium would become possible.

Looking forward in this field, the question was whether there were more transuranic elements to be discovered? All the isotopes beyond uranium were so short-lived they could only occur if they were continually generated. Not only was radioactive decay a source of instability but spontaneous fission became increasingly important as a limitation on the survival of nuclei as these become very heavy. It was believed that the limit was likely to be reached at about element 108.





★ **WHAT IS microchemistry, or micro-chemical analysis?** This question was put to me this week and although I answered it, I was pleased to hear the views of Mr. R. C. Chirside, chief chemist at the GEC research laboratories. To what does the micro refer? To the chemistry; to the sample; to the quantity or to the proportion of the element or substance to be identified or determined; to the size of the apparatus; or even, as has been suggested, to the stature of the analyst?

Mr. Chirside said that having studied the whole of the symposium programme he was no nearer a definition. In fact he declared himself a heretic for he has never been completely convinced that there is such a thing as microchemistry of microanalysis. After all, the inorganic analyst has been carrying out micro-chemical analysis for a very long time.

The organic microchemists, Pregl and Emich, and others who have followed, have of course carried out great pioneering work, as Mr. Chirside recalled and as was ably emphasised in the second Plenary Lecture given by Pregl's outstanding pupil and successor, Professor Hans Lieb. Now I learn there is growing up at Birmingham a sub-micro chemical analysis school.

★ **ON THE EVE** of the second Atoms for Peace exhibition at Geneva, Constructors John Brown have claimed that before long the West need no longer be dependent on the US for economic supplies of heavy water. CJB will offer, for an outlay of about £2½ million, a packaged plant that can be linked with any synthetic ammonia stream to produce D<sub>2</sub>O in quantity, at an all-in cost of about \$21.50 (£7 14s) per lb., of £17 per kg., as against the US list price of \$28 (£10) per lb. or £22 per kg.

Every country with nuclear power projects is anxious to produce and control its own supplies of heavy water and break the US monopoly which carries the condition that US technicians must have access to buyers' premises. In Germany, Hoechst are completing a pilot plant designed to produce 6 tons of D<sub>2</sub>O a year—from ammonia; Pintsch-Bamag are developing the US technique of ordinary water extraction, while Degussa are working on the Becker process, in which a liquid-phase exchange of deuterium between hydrogen and water is catalysed. In the UK, research at Harwell and the universities on the distillation of hydrogen at -230°C to extract deuterium that can be burned with oxygen to yield heavy water is being worked on by Petro-carbon Developments with a view to commercial exploitation.

Now CJB claim to have perfected a process that can be linked economically

to the nitrogenous fertiliser plants that are springing up all over the world. They state that as a rough guide, the capital cost will be in the region of £80,000 per tonne of heavy water per year. Operating costs depend largely on steam and power costs, but in representative European conditions, allowing 15 per cent to cover interest on capital and depreciation plus £5 per kg. for operation, the cost of heavy water should be about £17 per kg. Designed to extract 70 per cent of the deuterium in the gas treated and operating on an 8,000 hour year, the plant would yield 28 metric tonnes a year. Operating staff involves 16 men working a four-shift system, with an additional eight men on day labour and a plant superintendent.

★ **A SCOTTISH** twenty shilling piece, dated 1696 and bearing the head of William III, is one pound that Monsanto Chemicals Ltd. will never spend. Now reposing in a place of honour in a safe at Monsanto House, London, the coin was gained in exchange for 'one hot pressed naphthalene plant'.

The coin originated from an agreement made in May, 1952, when Monsanto realised they would need more naphthalene for phthalic anhydride production than seemed likely to be available from existing sources. It was then agreed that Scottish Tar Distillers should install and operate a new hot pressed naphthalene plant for at least five years, or until a production of 10,000 tons had been reached. After that, it was stipulated that the plant would become the property of STD on payment of £1 to Monsanto.

The 10,000 ton level was reached recently and to mark it, Sir Francis Tudsbury, STD chairman, presented Monsanto with the token £1. Monsanto representatives were delighted at receiving the historical twenty shilling piece, which was one of the last to be minted in Scotland before the Act of Union.

★ **A CHEMICAL** with an interesting future that has recently come into the news in this country is cyanuric acid. Two British firms are interested in the product, Whiffen and Sons and the Cocker Chemical Co. First in the field were Whiffens, one of the Fison group, who have been producing experimental quantities, but who are not yet ready for commercial production.

Two weeks ago CHEMICAL AGE published the news that the US Food Machinery and Chemical Corporation had come to an agreement with Cocker Chemical for the distribution of cyanuric acid in this country. The intention is that supplies should initially be imported, but that a UK production unit

should be set up as a joint venture by the two companies.

The potential market is big, but there must be some doubt as to whether it is yet large enough to support two major firms. The product is of interest for a number of reasons: for modifying synthetic resins, for use in chemical reactions, etc., and in the chlorinated forms as a dry bleaching agent, as an additive to synthetic detergents, scouring powders, etc. Cocker Chemical, of course, have much experience in the handling of chlorine. In the States, it has been developed greatly at the expense of dichlorodimethylhydantoin, which is marketed by Wyandotte as Halane and by Glyco Products as Dantoin. DDMH is made in the UK by Whiffen.

★ **THE MOST** comprehensive booklet yet published on fused quartz and silica is now being made freely available under the title 'About Vitreosil' by the Thermal Syndicate Ltd., Wallsend, Northumberland. This interesting and well-produced book is neither a catalogue nor a price list, but fills a gap for there is little published work on the subject.

Every aspect is treated in great detail that clearly bears the authority of the 50 years of research and development that Thermal Syndicate have put into the production of Vitreosil.

Brief mention is made of Spectrosil, a new synthetic fused silica, a material with total impurities of less than 1 p.p.m. This is available in the form of crucibles, boats, lenses, prisms, discs, tubes, rod and wool. It is said to be unaffected by exposure to gamma radiation and pile radiation and to have a much higher transmittance than fused quartz below 2,000Å.

★ **A MAN** of many parts is Mr. R. Barrington Brock, a director of Townson and Mercer, Croydon, who was recently elected president of the Scientific Instrument Manufacturers' Association. A Fellow of the Royal Institute of Chemistry, he was chief chemist of Ilford's Imperial Dry Plate Co. and was later associated with the development of Dufaycolour.

His experience has therefore been both as a user and maker of instruments. Another of his research activities is the growing of vines in the open. Mr. Brock has produced some fine wines from grapes grown in those conditions and has also produced brandy; he is in fact one of the select few who hold individual licences to distil alcohol in this country.

Perhaps his favourite hobby is the building and racing of fast cars. Mr. Brock drives a car that he designed and built himself which with a rear mounted Jowett Javelin engine that will give 107 m.p.h. and 42 m.p.g. Another of his more dangerous hobbies takes him to Switzerland in the winter sporting season to the Cresta run.

*Alembic*



# Birmingham Microchemistry Symposium

## Attracts More Than 400 Visitors

### Chemists from 24 Countries Hear World's Experts

**W**ELCOMING delegates to the International Symposium on Microchemistry held at Birmingham University from 20 to 27 August, the chairman of the Executive Committee, Mr. J. R. Leech, J.P., said that 24 countries were represented. Among the 420 delegates were most of the outstanding micro-chemical scientists throughout Europe and the world.

It was in 1954 that the Midlands Section of the Society for Analytical Chemistry had held a highly successful analytical symposium. This same section and the Microchemical Group of the SAC, on behalf of the SAC had organised the present symposium, under the patronage of International Union of Pure and Applied Chemistry.

Referring to the president of the symposium, Professor Maurice Stacey, Mason Professor and Head of the Chemistry Department, Birmingham University, Mr. Leech said Professor Stacey was well known for his work on carbohydrates and organic fluorine compounds. He was also one of the first to be interested in microchemistry.

Professor Stacey considered it was appropriate that the symposium should be held when Dr. R. Belcher, Reader in Analytical Chemistry at the University of Birmingham and chairman of the Midlands Section, SAC, was president of the Analytical Section of IUPAC.

In giving a special welcome to the plenary lecturers and overseas guests, Professor Stacey described Birmingham as the heart of industrial England and the centre of the Industrial Revolution. This country was now in the midst of another revolution—an atomic revolution. He liked to think that the analyst was quietly playing his part in it. Analysts had a great deal to be proud of today. He recalled that at one time when a person could not be fitted into sales, etc., they placed him in analytical work. Analysts today were the 'key-pin' in every sphere and their status had been raised.

**Professor Maurice Stacey, symposium president, welcoming members to the International Symposium on Microchemistry**



At Birmingham they were particularly proud of their analytical chemistry, and Professor Stacey announced that Birmingham University had been chosen for a £10 million expansion scheme. In the chemistry section, £1 million was being spent. Site preparation had begun for a new chemistry building with teaching laboratories, a microchemical department, more lecture theatres, common rooms and additional social facilities.

In the midst of these changes and great developments, considerable work was being carried on in developing analytical, micro- and sub-micro-analytical techniques. Medola medallist, Dr. T. S. West, was particularly concerned in studies on sub-micro techniques.

tion analysis. Mr. James then indicated some of the pitfalls and ambiguities that could arise taking silicon as an example and the extent to which radioactivation analysis met analytical requirements. The activation technique—activation, chemical separation and measurement were then considered.

**Activation:** Neutron activation was dealt with. The three chief reactions concerned were: the  $n\gamma$  reaction which required neutrons of thermal energies and produced an active species of the identical chemical properties to the parent atom. Then the  $np$  and  $nz$  reactions requiring neutrons in excess of a given energy (1 MeV) which gave rise to active species chemically different from the original impurity.

In addition to the  $np$  reaction on sulphur and the  $nz$  reaction on chlorine, there were consecutive reactions occurring with silicon. This latter reaction set a practical limit to the determination of phosphorous activity in silicon at 0.002 p.p.m.

It was considered that by alteration of the ratio of fast and slow neutrons, it should be possible to differentiate between these reactions. It was essential to have a large neutron flux to obtain activity. Available positions in the thermal column where fast neutrons were greatly reduced had insufficient flux available ( $10^{10}$  n/cm<sup>2</sup>/sec. in BEPO). It was believed however that the available positions in DIDO had sufficient flux with reduced fast neutron content. It was, of course, essential to have a standard sample irradiated at the same time for comparison with the sample for analysis, and the standard must not suffer from self shielding. Ways of minimising this effect were irradiation of dilute solution, spots on filter paper, etc. The author, however, preferred the use

## Determination of Trace Elements in Semi-conducting Materials

**W**ITH the need for a very high state of purity for elemental semi-conductors such as silicon and germanium, in devices such as high voltage rectifiers, a unit for impurity concentration-carriers/c.c. has been introduced. Mr. J. A. James (Research Laboratory, The British Thomson-Houston Co. Ltd., Rugby) discussing 'Determination of Trace Elements in semi-conducting Materials,' stated that intrinsic silicon would have a carrier concentration of 300° K of  $1.27 \times 10^{10}$  carrier/c.c. ( $n = p$ ) and taking ionised impurity atoms as equivalent to carriers then this would correspond to the impurity atom in  $10^{13}$  silicon atom or  $10^{-7}$

p.p.m. by atoms which was the same in p.p.m. by weight for aluminium and phosphorus (two important impurities) or  $3 \times 10^{-8}$  p.p.m. by weight for boron.

Intrinsic silicon would have a resistivity of  $2.4 \times 10^5$  ohm-cm. Silicon crystals grown at present rarely exceed 100 ohm-cm., that is, the impurity level was 200 times that described above. Equivalent impurity level in intrinsic semi-conductors was an exponential factor of the energy gap. Figures for germanium were 1,000 times larger than for silicon, while for silicon carbide there were  $10^{16}$  times smaller.

One of the few techniques capable of approaching these limits was radioactiva-



**Dr. C. L. Wilson (Queens University, Belfast), left, with his brother D. W. Wilson (John Cass College, London), centre, and Dr. Rudolf Pribil (Czech Academy of Sciences)**



General view of the assembly at the opening. Bottom left is Miss Daphne L. Mermikides, assistant editor of "Chemical Age"

of a solid which could be removed cleanly from the ampoule, was sufficient to weigh accurately and afforded duplication of the standard determination.

**Chemical Separation:** Specific steps should be taken to avoid radiochemical contamination of the separated fractions, e.g., Zn, P, and Cu. Zinc contamination was noted in a first attempt at determination of iron in silicon. Subsequently zinc carrier was added and separated using quinaldine acid reagent followed by the usual iron separation. Gamma spectrometry was a very useful tool here for a quick survey of an unknown sample before selection of a separation scheme. Where only a few counts above background were registered, further experiments were required to be confident of the purity.

Addition of carrier should be made at the earliest possible stage, it was emphasised. It was important to make sure that the carrier and the impurity were converted to the same oxidation state.

For determination of arsenic in silicon the method of solution was of great importance since if the sample was dissolved in HF/HNO<sub>3</sub>, then radioactive arsenic was lost although the arsenic added as AS<sub>2</sub>O<sub>3</sub> as carrier was not. Solution in aqueous NaOH/H<sub>2</sub>O<sub>2</sub> gave higher activities, although there was still some loss. The loss probably occurred as arsine in bubbles of hydrogen liberated by solution of the silicon matrix. Use of fused NaOH had reduced the likelihood of losses from this cause.

**Measurement:** It was necessary to see that the standard and the sample were in the same form and of approximately the same weight to compare the count, so eliminating absorption errors, especially for soft  $\beta$  emitters. Where the half life was long,  $\gamma$  spectrometry might help, although if the  $\beta$  count observed was low it would be below the detection limit for the  $\gamma$  spectrometer. Further separations with scavenge or hold back carriers for possible contaminants might be advisable until constant specific activity was obtained.

Detection limits varied widely from gold at  $10^{-11}$  to iron at  $10^{-6}$ . Some 17 elements had been determined in silicon. Gold and arsenic could be determined down to 1 in  $10^{11}$ . Boron, aluminium and oxygen could not be determined by radio-activation methods.

**Mass Spectrometry:** With the solid source mass spectrograph it was possible to determine boron down to 1 in  $10^7$ . Detection limit of the instrument had been tested by specially prepared doped samples of single crystal silicon and found to be 0.1 p.p.m. for boron to 0.01 p.p.m. for Sb. Some blind spots could arise due to over-lapping spectra. The method gave an overall picture of any unknown material similar to optical spectroscopy, but also covering non-metal. These were covered down to about 1 in  $10^7$ .

In theory the isotope dilution technique should be capable of much lower detection limits since the isotope ratio could be measured to 1 per cent on  $5 \times 10^{-8}$  gm. of boron. As an example of the limitations of this method determination of boron in silver was discussed. The usual chemical blank was required as the 'spiked' sample had to be processed to extract the impurity and be converted to a suitable form, e.g. sodium borate, for analysis in a surface ionisation mass spectrometer. The need for a quantitative yield was eliminated. A bromination reaction was under investigation. A low blank had been achieved but the yield was as yet too low in the sub-microgram range.

### Proceedings

Proceedings of the International Symposium on Microchemistry, which will include reports of discussions, will be published by Pergamon Press Ltd., 4 Fitzroy Square London W1.

## Use of Fungi to Determine Trace Metals in Biological Materials

A MICROBIOLOGICAL method was described by Dr. D. J. D. Nicholas (head of the Biochemistry Department, A.R.C. Unit of Plant Nutrition (Micronutrients), Long Ashton Research Station, University of Bristol) for determining minute amounts of Fe, Cu, Zn, Mn and Mo in biological materials using the mould *Aspergillus niger* (Mulder strain) as the test organism. The mould was stated to grow well in liquid culture containing glucose, the macronutrients N, P, K, Mg, Ca and P and the trace metals Fe, Cu, Zn, Mn and Mo. When one of the essential nutrients was withheld from an otherwise normal culture solution, the fungus did not grow well as shown by reduced dry weight of the felts and restricted sporulation.

Special chemical methods have been developed to remove the trace metals from the macroconstituents of the media since it has been found that even in the best grade materials there are sufficient trace metals present as contaminants to provide for the optimum growth of the fungus.

A standard series for any essential nutrient can be prepared by returning graded amounts of the test nutrient to 50 ml. lots of the purified culture media dispensed in 500 ml. Erlenmeyer flasks. The flasks inoculated with a spore suspension of the fungus are incubated in the dark for four days at 30°C. The growth of the fungus is proportional to the nutrient added over the deficiency to sufficiency range. Biological material is assayed for trace metals by adding known weights or volumes of it aseptically to 50 ml. lots of medium that was depleted of the test nutrient. Under these conditions stated Dr. Nicholas, the growth of the fungus will depend on the amount of the test nutrient it derives from the biological material added since there is none of it present in the culture solution.

### Minimal Amounts of Trace

The following minimal amounts of trace in  $\mu\text{g}$  could be determined with an accuracy of  $\pm 5$  per cent in biological materials:— Fe, 0.01; Cu, 0.05; Zn, 0.25; Mn, 0.01; Mo, 0.00001. The assay for Mo was the best known at the present time.

Microbiological assay of trace metals had been made in soils both from temperate and tropical areas. The results obtained were in closer agreement with the response of higher plants to trace metals than were those obtained by current chemical extractants of nutrients from soils. The method had also been used to determine trace metals in extracts and in the ash of plants. A novel use of this technique had been the demonstration that Mo concentrated in purified fractions of the enzyme, nitrate reductase, in plants. No other method was at present sensitive enough to determine accurately such low levels of Mo in the protein fractions.

Advantages of the bioassay method for trace metals included (a) a quantitative determination at levels that were often below the limits of present day chemical or physical techniques, (b) a direct method that involved the minimum preparation and dilution of the sample, (c) results for soils that reflected the trace metal deficiencies in higher plants. Disadvantages included the need for rigorous pure culture methods and the frequent use of standard series for the trace metals to



Dr. W. J. Kirsten (Uppsala University, Medicinsk-Kemiska Institution, Uppsala, Sweden) left, and Dr. Petr. Zuman (Czech Academy of Polarography)

check that a mutation had not occurred in the organism and that no residual chelating agent was left in the media after the purification procedures. Dr. Nicholas reported that the method had been used to good effect to determine quantitatively minute amounts of trace metals in soils, plants, and in enzyme preparations.

### Titrimetric Finish of Chromatographic Spots

A DIRECT titrimetric determination of fine trace elements on paper chromatographic spots was described by Professor Alice Lacourt (Microchemistry Centre, Brussels University, Belgium). The spots are not washed out as is usual and the reagents and apparatus required are to be found in any chemical laboratory.

The technique has been studied using five inorganic elements. It is stated to be perfectly applicable to determinations, organic substances on paper, after chromatographic separation.

The amounts of tungsten, vanadium cobalt, copper and zinc titrated were as low as a few micrograms. Reproducibility is governed by endpoint discrimination and reproducibility of the blank determinations, and is quite satisfactory down to the microgram level. Titrimetric finish is reported as accelerating the quantitative chromatographic procedure by suppressing elution.

The technique offers a simple aid to quantitative chromatographic investigations in the absence of a spectrophotometer. It does not, however, replace the spectrophotometric finish for determining spot concentrations on paper, when the amounts to be determined are below the microgram level and when the accuracy required should be better than 1 to 2 per cent.

## DETERMINATION OF SULPHUR HALOGEN, BY Dr. KIRSTEN

EXPERIENCES in the determination of sulphur and halogen were reported by Dr. Wolfgang P. Kirsten (Institute of Medical Chemistry, University of Uppsala, Uppsala, Sweden). He determined sulphur by dry combustion in oxygen and subsequent hydrogenation in another chamber of the same quartz tube.

The sulphur hydrides formed were absorbed in hot alkaline potassium iodate which oxidised them to sulphate. Excess of iodate was back-titrated with thiosulphate. Dr. Kirsten said that nitrogen and halogens did not interfere. Interference by phosphorus was negligible in micro-analysis.

From ash-containing compounds the sulphur was expelled with tungsten trioxide. The combustion-hydrogenation method was indicated as being very suitable for ultramicro- and trace-determination of sulphur. For determination of halogen Dr. Kirsten said that he had modified the oxygen-flask combustion method of Miki and Pech-Schöniger for the analysis of volatile liquids which were weighed out in thin polythene tubing.

### Discussion Session

Mr. G. Ingram (Courtaulds Ltd. Berks) asked what were the advantages of using hydrogen. The reducing method had been dropped, in favour of the oxidising method. Now there appeared to be a combination of the two. Mr. Ingram also wanted to know about the risk of explosion. With regard to his own experience with sulphide, he had found the iodate method extraordinarily good and in his opinion this was the best method of estimating sulphide.

Dr. Kirsten stated that the apparatus for sulphur absorption required strong agents.  $\text{SO}_2$ ,  $\text{SO}_3$ ,  $\text{Cl}_2$  and F were very difficult to absorb. But if these were hydrogenated  $\text{H}_2\text{S}$ ,  $\text{HCl}$ ,  $\text{HF}$  were obtained which were easily soluble in

water. Also, there were few interfering ions.

With regard to the risk of explosions, Dr. Kirsten said that he had used the combustion method he had described for eight years without an explosion. He said the three-way stopcock used in his apparatus prevented the gases (H and O) meeting. As a precautionary measure safety goggles were used for a day or two by beginners.

Professor F. Feigl (of the Ministerio da Agricultura, Lab. Prod. Mineral, Rio de Janeiro, Brazil) asked the lecturer about interference by arsenic or antimony.

Dr. Kirsten thought that arsenic would form a mirror on the combustion tube.

Dr. A. Steyermark (Hoffmann-la-Roche Inc. Nutley, New Jersey, US) asked Dr. Kirsten whether he was going to expand his method for fluorine.

Replying to this inquiry, Dr. Kirsten said he had not yet had occasion to take much interest in fluorine.

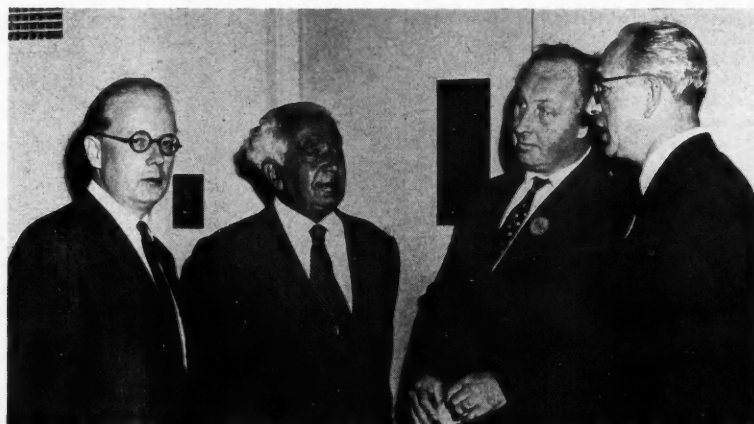
Mr. L. Blom (The State Mines, Netherlands) was interested in the measurement of chlorine during combustion. He had used the Schöniger method but had had difficulty with high chlorine contents. He thought that Dr. Kirsten's use of polythene tubes for the samples was excellent and would overcome present difficulties.

Miss M. Corner (DSIR, Chemical Research Laboratory, Teddington) said that she had found that in the case of fluorine and chlorine determined by the Schöniger method if carbon was present, no further trouble was experienced.

Mr. G. Ingram said that sodium bisulphite and caustic soda was good for effecting solution.

Dr. Kirsten admitted that his results with bromine and chlorine had not been very good.

Dr. R. Levy (Microanalysis section,



At the opening session, l. to r., Dr. R. Belcher, reader in analytical chemistry, Birmingham University; Prof. Dr. Fritz Feigl, head of microchemical department, Laboratoria da Produção Mineral, Rio de Janeiro; Professor Maurice Stacey, Mason Professor of Chemistry, Birmingham University; and J. R. Leech, chairman of the symposium executive committee





L. to r., J. H. W. Forsythe (Queens University, Belfast), Dr. Rudolf Pribil (Analytical Laboratory of the Czech Academy of Sciences), Dr. J. Magee (Queens University) and Mrs. Magee

Laboratoire Municipal de Paris, France) said that the Br ions might not be in the form desired. For a solution in alkaline medium, hydrogen peroxide had been used by him with good results.

Dr. Kirsten reported that that was what he had done. He had used 0.5 ml. of 30 vol. hydrogen peroxide but had still had no success.

Dr. Levy said he had found it necessary to boil the solution for 15 minutes.

Dr. Kirsten thought this might well be the solution for he had only been boiling for 10 minutes.

Mr. E. E. Archer (Epsom, England) said that with regard to absorption of chlorine he used  $\frac{1}{2}$  ml. saturated sulphur dioxide solution. Absorption was complete in an hour. He titrated chlorine as hydrochloric acid.

## Rapid Microdetermination of F, Cl, and S in Organic Compounds

A SERIES of investigations has been carried out by Dr. Roger Levy (Micro-analytical Centre of the Municipal Laboratory of Paris, Paris, France), limited to the micro-analytical field, using the oxy-hydrogen micro-combination and the related titrimetric methods of determining the halogens and sulphur. These investigations have resulted in the design of an apparatus and in a procedure applied to the microdetermination of fluorides, chlorine and sulphur in organic compounds.

In principle, the sample is subjected first to a thermal treatment to transfer its constituent elements into the gaseous phase in the form of distillation-, pyrolysis- or combustion gases. These gases enter the flame of an oxy-hydrogen blast micro burner, burning in an enclosed space, where the elements for analysis (halogens and sulphur) are converted to inorganic molecules (halohydric acids, sulphurous anhydride), which may be determined. Water vapour produced by the blast burner is condensed in a cooling device; the halohydric acids and sulphurous anhydride are more or less partially retained in condensed water; their quantitative retention is eventually completed in an absorption device containing a suitable reagent.

To exclude ignition of the oxy-hydrogen blast burner in surrounding air, a blast burner consisting of a tube of trans-

Dr. J. Haslam agreed with Mr. Archer about bringing down bromine by this method. He inquired whether Dr. Kirsten was 99 per cent successful. With regard to iodine compounds, he wondered whether Dr. Kirsten had tried iodine compounds which did not contain sulphur.

Replying to Dr. Haslam, Dr. Kirsten said that he employed back titration, using 10 ml. solution. He had not detected interference from iodine. It was, of course, difficult to detect iodine after titration with 10 ml. of solution and back titrating with a similar amount.

Mr. Ingram considered that iodine would not interfere.

Dr. Kirsten then asked about iodate, and Mr. Ingram felt that iodate would not interfere, but it would depend upon concentration; with which Dr. Kirsten agreed.

parent silica (molten and blown quartz) was used, which was placed horizontally and composed of two parts welded together. The first part is comparable to a classic combustion tube, with a lateral inlet for gas and ending with a 10 mm. long by 5 mm. dia. capillary tube.

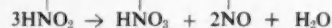
Thermal treatment takes place in the front part of the tube with circulating oxygen. Combustion gases and excess oxygen flow through the capillary tube into a straight tube where hydrogen is introduced. A fine stream of oxygen can be injected into the atmosphere of hydrogen. It has been found sufficient to heat the second part of the tube externally below the capillary by Bunsen burner or an electrical furnace to produce ignition of the blast burner. If ignition is equal to at least 850°C at the moment of oxygen injection, and if this injection is instantaneous, explosion of the mixture does not occur.

Feeds of oxygen and hydrogen in the blast burner can be reversed and the gases evolved by thermal treatment of the sample can be transferred either in oxygen or hydrogen. In the case of the latter preliminary heating of the sample may be advantageous. Only the oxidising flames or approximately stoichiometric ones (producing a 'neutral' flame) are able in general to obtain a quantitative mineralisation.

During blank tests, adjustment of the

flame to an 'oxidising' or 'reducing' flame frequently produces alkaline or acid condensation water. Nitrogen in the oxygen is responsible for this phenomenon, producing ammonia with a reducing flame and nitrogen oxide in an oxidising flame. The gases fed into the burner are compressed to 250 kg. Electrolytically-produced hydrogen from chlorides should be avoided as it may contain chlorine.

The presence of nitrous acid can affect subsequent titrations, in particular by its slow decomposition thus:



falsifying the titration of F<sup>-</sup> ions if these are determined by the indirect alkali titrimetric finish. Nitrous acid should be decomposed by boiling with hydrogen peroxide.

Water vapour produced carries on the combustion products to be determined and is condensed by continuous cooling effected by a refrigeration exchange. Full details for cooling were given by Dr. Levy.

### Determination of Fluorine

In the case of microdetermination of fluorine, the hydrofluoric acid formed is retained quantitatively due to its very high solubility and relatively high boiling point (19.4°C). In the case of HF the collecting tube is of transparent silica as is the collecting titration beaker. When rinsing is finished, the fluoride ions can be titrated directly in the beaker.

In the case of chlorinated substances or those containing sulphur, to fix the hydrochloric acid or sulphurous anhydride, the combustion gas in the case of HCl is bubbled into a small volume of distilled water and sulphurous anhydride is fixed by bubbling into aqueous soda solution with diluted H<sub>2</sub>O<sub>2</sub>. A special absorption vessel of Pyrex glass has been designed for this purpose which can be used as a titration flask.

To compensate for losses of pressure in gas flow (i.e. due to the flow of gas through the sintered glass plate, and to immersion depth in the retaining liquid) a vacuum apparatus is connected to the outlet tube of the ground stopper of the collecting flask.

For the two external sources of heat required for the oxy-hydrogen combustion, a simple Bunsen burner is adequate for the ignition of a blast burner and for precombustion a suitable micro-burner. To avoid contamination of the surrounding atmosphere, however, electrical heating devices are used. The heating element described by Dr. Levy consists of ceramic tubular support with a lateral window, and notched with 19 small slots on each terminal section which support resistant heating wire (0.8 mm. dia. Kanthal). This element is inserted in a cylinder of stainless steel. Asbestos provides the insulation. The sample is burned by direct radiation from the parallel portions of exposed wire which is maintained at red heat. The furnace igniting the blast burner is placed on the right side and the combustion furnace is on the left side.

Intensity of heating current for the ignition furnace is 9 to 10 amp. (45 volts) and produces a temperature of 850°C in the tube axis within three minutes. The combustion furnace temperature is about 350°C at the level of the boat within 1 m. 30 sec. using a heating current of 4 to 5 amp. (20 volts), and intensity of heating is increased up to 9-10 amp. when the boat temperature reaches about 950°C after four minutes.

Gas feeding is controlled by an industrial reduction valve with a membrane producing the first step of expansion to a pressure of about 1,000 gm./cm<sup>2</sup>. A second similar type reduction valve with a membrane having a diameter of 10 cm. (double that of the first) controls the working pressure. All connections and intermediary joints between reduction valves, flowmeters, capillary tubes and combustion tube are made with polyvinyl chloride tubing.

After ignition of the blast burner, the sample boat has to be moved 30 mm. from the opening of the capillary in order to carry out pre-combustion. This operation is performed by means of a magnetic pushing device (cylinder of soft iron encased in glass) driven by an external magnet moving parallel to the combustion tube.

### Explosion Safeguard

For protection from explosion the apparatus is installed in a support of sheet iron. Compartments for combustion, and condensation and rinsing are separated by an asbestos screen, and are screened in front by Plexiglas 8 mm. thick sliding vertically.

The operating technique is to be described by Dr. Levy in a paper still to be published.

#### Essential Times of Combustion

Operation	Time
Ignition of burner ... ..	3 min.
Insertion of boat ... ..	30 sec.
Heating sample (to 350°) ... ..	1 min.
Heating sample (to 950°) ... ..	4 min.
Rinsing (with heating) ... ..	5 min.
Rinsing (without heating) ... ..	1 min.
Cooling between two analysis ... ..	1 min.
<b>Total Time</b> ... ..	<b>16 min.</b>

Some substances (e.g. sulphanilamide) produce a carbonised deposit in the capillary of the burner. To avoid this the ignition furnace must be placed over the capillary for the first six minutes of combustion.

**Micro-determination of Fluorine:** Oxy-hydrogen combustion of a substance containing fluorine provides directly a solution of hydrofluoric acid in a volume of about 50 ml. in a silica beaker. The method of titration is based on the determination with indirect alkalimetry (Dehal, Levy and Moureu, *Microchim. Acta*, 1957, 396). Nitrous acid is decomposed by neutralising the HF solution with 0.1 N soda then acidifying with 0.05 N HCl in presence of two drops methyl red-bromo cresol green (pink colour). After adding 30 per cent H<sub>2</sub>O<sub>2</sub> (0.25 ml.) the solution is boiled for 5 min. The neutralised solution (mixed indicator now grey-violet) is then titrated with indirect alkalimetry.

A series of fluorine micro-determinations of organic samples at the rate of ten determinations per normal working

day and per person, with weighings carried out by someone else, can be effected. The method has been tested by the analysis of the following: tri-fluoro-acetanilide (1), m-trifluoro-methyl-benzoic acid (2), p-fluoro-benzoic acid (3), polytetrafluoro-ethylene (Teflon) (4), and perfluoro-dicyclo-hexyl-ethane (5). The weight of samples varied from 2 to 3 mg. for 4 and 5 and from 3 to 6 mg. for substances 1, 2 and 3.

Accuracy of the total analytical procedure is high. This has been determined by calculation of the typical difference  $s_F$  between the weight of fluorine found and weights of fluorine truly present in the samples. For the total analysed substances:  $s_F = 8 \mu\text{g}$  of fluorine, i.e.  $0.4 \times 10^{-16}$  of fluorine equivalents, has been found.

The solutions are obtained by combination of distilled water introduced initially in the absorption flask (8 ml.), condensed water and washing water from the tube with the sintered glass plate (total about 30 ml.). Nitrous acid is removed (by boiling 10 minutes with 1 drop HNO<sub>3</sub> (d = 1.40) and 0.25 ml. 30 per cent H<sub>2</sub>O<sub>2</sub>).

Potentiometric argentometry is used for the titration of chlorine (as chloride) (Levy, *Bull. Soc. Chim.*, 1956, 497).

**Micro-determination of Chloride:** Serial micro-determination for chlorine can be performed at the rate of at least 12 complete determinations per normal working day, and per person.

The procedure has been tested by the analysis of the following: chlorhydrate of benzyl iso-thiourea (1), chloro-I diniro-2,4-benzene (2), p-chloro-nitro benzene (3), o-chloro benzoic acid (4), p-dichlorodiphenyl sulphone (5), polyvinyl chloride (6), pentachlorophenol (7) and pentachloroethane (8). Weights of samples varied from 3 to 7 mg. for substances 1 to 4 and from 2 to 4 mg. for substances 7 and 8.

Accuracy of the procedure has been determined by the calculation of the typical difference  $s_{Cl}$  between weight of chlorine found and weight of chlorine present in reality in samples (1) and (5). The value of the difference is:  $s_{Cl} = 6 \mu\text{g}$  Cl, i.e. about  $0.17 \times 10^{-6}$  of chlorine equivalents.

**Micro-determination of Sulphur:** An oxidising procedure in the combustion of sulphur compounds, for absorption an alkaline washing liquid is used in com-

bination with an oxidising agent (3 ml. 0.1 N soda and about 1 ml. 30 per cent H<sub>2</sub>O<sub>2</sub>) of total volume 8 ml. Minimum bubbling of gas in the flask is required. Sulphur is determined in the form of sulphuric ions. To decompose nitrates, the solution is neutralised and slightly acidified with 0.2 N HCl in presence of a drop of sodium alizarin-sulphonate indicator (indicator used for final titration), are boiled for 10 minutes.

The method of titration of sulphuric ions used, is based essentially on the determination of SO<sub>4</sub> ions in an aqueous alcoholic medium of a controlled pH, by means of a standard solution of barium chloride in presence of sodium alizarin sulphonate as an absorption indicator (Le Peintre and Richard, *Chim. Anal.*, 1957, 39, 331). Every titration should be carried out immediately after the corresponding combustion.

An operation can carry out 12 determinations per normal working day. The method has been tested by analysis of p-dichloro-diphenyl sulphone (1), chlorhydrate of benzyl-iso-thiourea (2), sulphanilamide (3) and thiourea (4). Sample weights varied from 3 to 6.5 mg. for substance 1 to 3 and 2 to 3 mg. for substance (4). The results are described as most satisfactory. Typical difference  $s_S$  between contents of sulphur found and contents truly present in the samples varies from 6 to 8  $\mu\text{g}$  of sulphur (i.e.  $s_S = 0.22 \times 10^{-5}$  of sulphur equivalent) while the typical difference of the titration amounted to about 2  $\mu\text{g}$ .

**Simultaneous micro-determinations:** Results of simultaneous micro-determinations on one sample indicate that oxy-hydrogen micro-combustion may be easily applied for this purpose.

In an investigation of 'Mostafion'—consisting of poly-trifluoro-chloroethylene (48.94 per cent fluorine and 30.44 per cent chlorine) combustion is effected with excess of oxygen. Two analytical procedures have given interesting results. The first includes titration of Cl<sup>-</sup> ions by potentiometric argentometry; then filtration of the titrated solution using a sintered glass No. 2 filter, covered by 5 mm. asbestos fibres; rinsing precipitated silver chloride with warm water. The filtrate is concentrated in basic medium and F<sup>-</sup> ions determined with indirect alkalimetry. Results obtained so far described as excellent for chlorine as well as fluorine.



At the Birmingham symposium, l. to r., Dr. W. Schoniger (Sandoz, Basle), Dr. E. A. M. F. Dahmen (Koninklijke/Shell Laboratorium, Netherlands), L. Blom (Dutch State Mines), P. Gouverneur (Koninklijke/Shell), Dr. S. Holzel (Austria) and Professor E. Knappe (Institute of Microbiology, Jena, Germany)

Using the second procedure aliquots of the combined rinsing and absorbing liquids are taken and Cl<sup>-</sup> ions are titrated in one aliquot and F<sup>-</sup> ions in the other. Titrations are carried out separately with each fraction and the results are calculated in respect of the whole solution. To compensate for the division of the original solution the sample is increased accordingly to 6-7 mg.

Simultaneous determination of fluorine and sulphur is adversely affected by the necessity of adjusting oxy-hydrogen blast micro-burner to an excess of oxygen, required by the presence of fluorine. Sulphur requires adjustment as close as possible to stoichiometric conditions. Difficulties are probably due to too rapid a flow of gases carrying the anhydride. An efficient system of absorption may solve this problem.

For chlorine-sulphur determinations, the analytical procedures are the same as in the simultaneous determination of chlorine and fluorine. Tests have been carried out with benzyl iso-thiourea chlorhydrate and *p*-dichloro-diphenyl sulphone.

### Discussion

Dr. W. J. Kirsten (Uppsala University, Medicinsk-Kemiska Institution, Uppsala, Sweden) asked Dr. Levy whether there was any possibility of a reducing flame if the sample used oxygen during combustion.

Dr. Levy said a reducing flame could be used. Ammonia would therefore be formed and could be got rid of by alkali.

Mr. P. Gouverneur (Koninklijke Shell, Netherlands) wanted to know whether Dr. Levy had tried to determine chlorine and bromine simultaneously.

This determination had been tried, said Dr. Levy, after destruction with peroxide. Results with silver nitrate were good. However, large quantities of sodium thiosulphate were required.

Mr. Gouverneur inquired whether precipitation of one halogen should be accomplished before the second halogen was precipitated. Dr. Levy said no, one had to correct for this.

Another delegate wished to know how long a combustion tube lasted. Dr. Levy said his tubes had lasted for a long time. They were usually broken by being dropped, etc.

Dr. Levy was asked what happened if one wished to examine an inorganic and organic compound. Depending on the substance, it was possible to determine halogens, etc., in such a compound, stated Dr. Levy.

Dr. Kirsten said that with regard to the length of life of combustion tubes, in his experience and using a reducing flame, his tubes lasted for at least 3,000 tests.

Replying to a question on the length of time that combustion took, Dr. Levy indicated that combustion must take a certain length of time. The temperature had to rise from the pre-heat treatment stage of 350°C to combustion temperature around 950°C. It was important to raise the temperature over a period of time.

flow rates of 0.1 and 0.3 cm./min. was used. For the anion exchange; Deacidite FF collecting between flow rates of 1.1 and 0.62 cm./min. was used. 1 M  $\alpha$ -hydroxy-iso butyric acid eluant was used for the rare earth separation. Stock solutions of this eluant were made up having acidities of pH 2.45 by 0.05 steps to pH 3.3 concentrated ammonia being used to make any necessary pH adjustments. Added detergent kept drop size to 5  $\mu$ l.

Volumes of solutions have been sufficiently small to enable their direct application to the ion-source of a mass-spectrometer. Examples of the separation from their mixtures of selenium and tellurium, and of cadmium, palladium and tin, using anion-exchange micro-columns were described.

For separation of selenium and tellurium from other fission products on a microgram scale, it has been found convenient to separate and purify them on an anion exchange micro column. Selenium passes through the column in 10M HCl containing 1 per cent HNO<sub>3</sub>, but tellurium is strongly held; it can be washed off with water. (Flow rate was maintained at 10  $\mu$ l/min.) Cadmium, palladium and tin have been found to be strongly absorbed on to the anion-exchanger from 2M HCl containing 1 per cent HNO<sub>3</sub>; cadmium is removed by water, palladium by dilute ammonia and tin by 3M sodium hydroxide solution.

### Apparatus for Control of Column Separation

An electronic apparatus for the control of the column separation of radioactive mixtures was illustrated, and a device giving continuous automatic pH change in the eluant used on cation-exchange micro-columns was detailed.

Detection of activity in the column effluents was carried out by means of an active monitoring device. Essentially two EHM2 Geiger-Müller thin window counter tubes were mounted, each in a lead castle 0.5 in. thick on each side of the thin-walled capillary tube.  $\beta$ - $\gamma$  radiation reached the counters through slit openings 1 mm. wide by 5 mm. long, and the output reached a rate meter and recorder.

The continuous automatic pH device consists of the micro column filled with an internal glass hook from which is suspended a 5 cm. glass tube sealed at the bottom except for a hole 100  $\mu$  in diameter near the bottom. This hole is stopped up with the end of a cotton thread, the tube filled with eluant of high pH and the reservoir with eluant of low pH. Levels in the tube and reservoir are adjusted to be equal. The cotton thread is pulled out and a tiny piece of Ferroxcube covered with polythene is dropped into the reservoir. The Ferroxcube stirrer is moved up and down by an electro magnet. Nitrogen pressure is applied to the reservoir. The pH of the column effluent is measured using a hanging-drop glass electrode.

## SEPARATING FISSION PRODUCTS ON ION-EXCHANGE COLUMNS

ION-EXCHANGE techniques have been applied by E. A. C. Crouch and I. G. Swainbank (UK Atomic Energy Authority, Chemistry Division, Harwell, Berks, to the separation of microgram quantities of fission products on very small columns. The complete separation of all the rare earth elements has been achieved on a cation-exchange micro-column, the separation being controlled by using induced radioactivity, and also by detecting visually the separated elements as oxalate precipitates.

Following results obtained with milligram quantities using 1 mm. diameter Deacidite FF in spheres and a column 25 cm. long by 5 mm. diameter, it was found that for microgram separations a column 2 cm. long and 0.8 mm. diameter filled with Deacidite FF in spherical particles of 34 $\mu$  mean diameter could be used. The column could be made smaller, but the anion-exchange resins allow separate elements to be isolated in the required concentration using the larger column.

The ion-exchange column used for the rare-earth separations described by Crouch and Swainbank consisted of the female part of a spherical glass joint mounted on a reservoir 7 cm. long, of 1.2 cm. diameter glass tube, the other end of which had been drawn to a cone

and attached to a 2.5 cm. length of thick-walled capillary tube (outside diameter 5 mm. and internal diameter 0.4 mm.). The end of the thick-walled capillary tube furthest away from the reservoir was sealed. A small bulb was blown in the capillary tube that could be pulled out to form a thin walled capillary tube 15 cm. long (outside diameter 0.7 mm. and inside diameter 0.2 mm.). The authors stated that the diameter of the capillary could be reduced if the activity of the elements to be separated was high and all the rare earths were of the same order of activity. The free end of the thin-walled capillary was cut of square. Nitrogen pressure was applied, and the capillary was dipped into dimethyl dichlorosilane, withdrawn, excess liquid removed and the tube dipped in pure alcohol. The tip was then water repellent and the size of the drop of water was about 10  $\mu$ l. The bottom of the thick-walled capillary was bridged with a layer of cotton fibre floated on to glass fragments placed in the bottom of the tube. Zeosark 225 and Deacidite FF (particle size less than 50 $\mu$ ) were used. Cylinders of 50 cm. length and diameters of 3.4, 4.6, 7.0 and 9.2 cm. were arranged as a series. For cation exchange separation of the rare earths, the fraction collecting between



# Method was Developed for Use with Mass Spectroscopy

Mr. R. G. Desmond (UK Atomic Energy Authority) asked whether any particular difficulties arose in steps leading up to application on ion-exchange column, especially with regard to knowing overall chemical yields on such small quantities.

Mr. Crouch said the method was developed to go with the mass spectrometer where the yield is determined by isotope dilution. The only requirement therefore was that sufficient material was recovered to put on the wire of the mass spectrometer. Standardisation of the isotopes was usually carried out by micro-titration, in which as little as 10 micrograms of a rare earth element can be titrated by means of EDTA with a standard deviation of  $\pm 1$  per cent.

Dr. J. A. Hunter (UK AEA) wished to know whether with radiometric assay of the eluate there was not a risk of the leaks indicating not complete elution of a particular element, but only completeness of elution of detachable material, with some small part of the element remaining absorbed or otherwise held in the resin bed.

Replying to this question, Mr. Crouch said he had no experience of this occurring. The rare earth elements were normally placed on the column in pure solution. If elution were carried out with nitric acid difficulties were sometimes experienced, but never where the eluant was hydrochloric acid. Generally the total rare earths added to the column were equivalent to several hundred microcuries of activity and after elution virtually no activity remained on the column.

Dr. R. J. Magee (Queens University,

Belfast) wondered if the operation of the micro-columns for separation of the rare earth elements was being carried out under equilibrium conditions. It would appear from the very slow rate of elution and the size of the column that equilibrium conditions were important factors if the separations were to be achieved. From the elution curves which had been shown it would appear that the distribution coefficients of the individual elements were very close together and conditions of separation would be very critical.

Mr. Crouch replied that Dr. Magee had literally hit the nail on the head. It was essential to work under equilibrium conditions. These conditions were chosen by reference to past work and separation was carried out under conditions where the column has the maximum number of plates and it is essential to choose flow rate consistent with the maximum plate efficiency of the column.  $\alpha$ -Hydroxybutyric acid was used as eluant because the separation factor for the various elements is high and equilibrate with the column rapidly at room temperature.

Dr. Magee then asked if the lecturer found any difference in the rate of exchange of the elements between cation and anion-exchange resins. In work in which small columns had been used it had been found that exchange with anion exchange resins was more rapid than with the cation exchange resins.

Mr. Crouch said he had no experience of this. He never had cause to measure the rate of exchange. In the case of anion exchangers one normally works far from equilibrium conditions due to their specificity.

volume of water was reduced by evaporation, in the presence of inactive carriers, and normal radiochemical procedures were used in the chemical separations. Cerium was obtained free from other activities by a series of fluoride, iodate and oxalate precipitations. Strontium and barium were separated from other elements by successive precipitations with fuming nitric acid and the barium (and radium) removed as chromate. Caesium was precipitated as the bismuth iodide complex and converted to the platinum chloride for counting. A preliminary separation together with potassium as the cobalt nitrites was sometimes included.

Determination of radiostromium in soil, vegetation, cereals, milk and bone was outlined by these workers. The calcium and strontium were extracted from the ash of vegetation, cereals and milk by treatment with acids, the calcium and strontium precipitated as phosphate and the strontium purified by successive nitric acid separations. Bone ash was treated directly by nitric acid and the strontium separated as nitrate. Three methods for determination of radiostromium in soil were described—by fusion with sodium hydroxide and sodium carbonate, by extraction with hydrochloric acid and extraction with neutral ammonium acetate solutions.

## Two Types of Counters Described

The activities were counted using end window GM tubes with a shield of cosmic ray tubes connected in anticoincidence. Two types of counters were briefly described, an automatic counter with a background of one count per minute and a counter suitable for very low activities such as are found in human bones with a background of 0.4-0.5 counts per minute.

An idea of the precision obtainable in the method as applied to rain water samples of average activity was obtained by splitting a very active sample into five aliquots, each of which was analysed by a different operator. Coefficients of variation for  $^{90}\text{Sr}$ ,  $^{89}\text{Sr}$ ,  $^{137}\text{Cs}$  and  $^{144}\text{Ce}$  were found to be about 5 per cent in each case. Results of independent examination of some samples by the New York Operations Office of the US Atomic Energy Commission were in good agreement with those of the UK laboratories.

Accuracy of the  $^{137}\text{Cs}$  determination was checked by counting an aliquot of a more active rain water sample on a low background gamma spectrometer with a 100 channel kicksorter. The result was 19  $\mu\text{Ci}$  compared with 18  $\mu\text{Ci}$  found by radiochemical method.

These results shown in Table I are those for the specific gravity of  $^{90}\text{Sr}$  in rain collected at Milford Haven. The season variation of the concentration of radioactivity in rain is illustrated in Figure 1.

Results obtained for strontium 90, from six sites in the UK were also shown

## Determining Fission Product Activity in Water and Biological Materials

THE world wide deposition of fission products from nuclear explosions and the relative importance of  $^{90}\text{Sr}$  were briefly discussed by Dr. F. J. Bryant, Mr. G. S. Spicer and Mr. R. G. D. Osmond (UK Atomic Energy Authority,

Research Group, Woolwich Outstation, Woolwich SE18).

Details were given of the techniques employed for the determination of  $^{90}\text{Sr}$  and  $^{89}\text{Sr}$ ,  $^{141}\text{Ce}$  and  $^{144}\text{Ce}$ ,  $^{140}\text{Ba}$  and  $^{137}\text{Cs}$  in rainwater and tapwater. The

TABLE I  
90 Sr Results Obtained by Different Laboratories

	A	B	C	D
Hay	19.5, 19.8	20	—	19.7, 17.9
Sheep bone	5.6, 5.6	5.8, 5.1	4.45, 5.14	6.98, 4.45
Milk—(1)	3.1, 3.0	3.3, 3.1	2.4	2.4, 2.6
Milk—(2)	4.85	4.3, 4.4	—	4.6
Human bone	1.12	1.37	—	1.24
Soil, HCl extraction	225	230, 217	—	—
Soil fusion	230	210, 218, 245	—	—
		243, 215	—	—

A. Woolwich Outstation, Chemistry Division, AERE.  
B. US Atomic Energy Commission, Health and Safety Laboratory, New York.  
C. E. A. Martell, University of Chicago.  
D. Nuclear Science and Engineering Corporation, Pittsburgh.

TABLE II  
Results for 90 Sr in Various Samples Collected in the UK

	Rainfall inches	Soil $\mu\text{Ci/gm}$	Grass $\mu\text{Ci/gm Ca}$	Sheep Bone $\mu\text{Ci/gm Ca}$	Milk $\mu\text{Ci/gm Ca}$
Omystwyth, Cardigan	60	11,500	1,350	200	32
Vyrimy, Montgomery	62	8,700	150	46	—
Princetown, Devon	81	14,500	150	5	17.8
Rockhope, Durham	42	6,100	650	100	18
Norwich, Norfolk	26	5,350	80	11.7	—
Boxworth, Cambridge	22	3,550	70	7.4	—

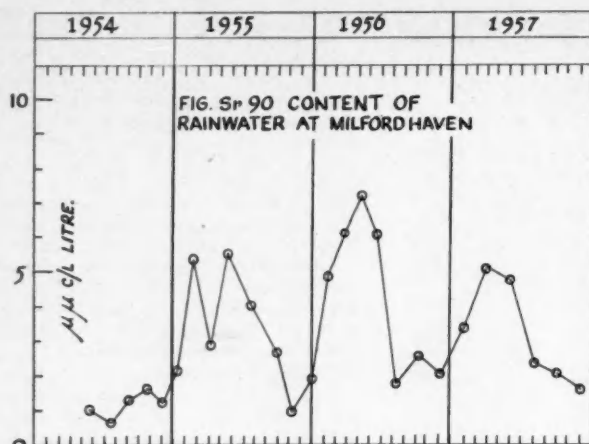


FIG. Sr 90 CONTENT OF RAINWATER AT MILFORD HAVEN

by Dr. Bryant *et al.* (Table II). Activity in soil was expressed as  $\mu\mu\text{c}$  per square metre and that for grass, sheep bone and milk in terms of the associated calcium.

The figures for milk from Wales, Welsh Border and Devon were well above average; those for southern and eastern England were about 5 to 6  $\mu\mu\text{c}/\text{gm}$ . Ca.

## Recent Developments in the Use of Ring-Oven Techniques

RECENT developments in the ring-oven technique were considered by Dr. Herbert Weisz (University of Technology, Vienna VI). The apparatus, he said, had been altered slightly, the ring-oven having a base-plate and a small electric lamp below the heating block. This allowed the edge of the heating-block to be seen, and so ensured better control of washing out.

Whereas in 1954, there was a systematic scheme for the analysis of 14 cations contained in one single drop of about 1.5  $\mu\text{l}$ , the scheme had been extended to include As, Ag and Hg.

Separation and identification of molybdenum and tungsten in one single drop had also been achieved by L. C. F. Blackman. In this instance a drop was placed in the centre of the filter and carefully 'ringed' and spotted with quinine hydrochloride to fix the tungsten. Unfixed molybdenum was washed into the sharp ring-zone on the ring oven with 0.1N HCl. After drying the inner spot containing the tungsten-precipitate was punched out. Molybdenum was identified by spraying with potassium thiocyanate and stannous chloride; a brick-red ring of Mo (III) thiocyanate complex formed. The punched out filter disc was placed in the centre of a fresh filter paper and all the tungsten was washed into the ring-zone with 0.1N ammonia solution and developed with Rhodamin B.

A purely qualitative application which was only possible on the micro-scale using the ring oven, was the detection of very small quantities of fluoride.

A drop of the blue solution of the complex aluminium with triphenyl methane-dyestuff Chromazurol S is placed on the centre of a filter paper and dried. The test drop is placed on

this spot and dried. The fluoride-ion forms the strong Al-F-complex, releasing part of the red Chromazurol S, which is marked, however, by the dark blue spot. The filter is placed on the ring oven and the red Chromazurol S is extracted with alcohol, forming a red ring if F<sup>-</sup> is present. Sensitivity claimed for this test is 0.005  $\mu\text{g}$ . If more F<sup>-</sup> is present, the rings are more intense. The method might therefore be a suitable semi-quantitative one.

**Ring-oven applied to anions:** A number of anion reactions have been tried by Dr. Weisz—eg, Br<sup>-</sup>, I<sup>-</sup>, BrO<sub>3</sub><sup>-</sup>, 10<sub>3</sub><sup>-</sup>, CNS<sup>-</sup>, Fe(CN)<sub>6</sub><sup>4-</sup>, Fe(CN)<sub>6</sub><sup>3-</sup>, S<sup>2-</sup>, SO<sub>3</sub><sup>2-</sup>, SO<sub>4</sub><sup>2-</sup>, NO<sub>2</sub><sup>-</sup>, NO<sub>3</sub><sup>-</sup>, CrO<sub>4</sub><sup>2-</sup>, PO<sub>4</sub><sup>3-</sup>. A scheme for several anions was now being worked out.

Some work had been carried out on spot-colorimetry in the field of semi-quantitative analysis. Statistically it has been shown that when 3 rings were made from varying numbers of drops of the unknown solution and compared with the standard scale—accuracy was better than  $\pm 5$  per cent. This would be quite satisfactory on a sample of 1-2  $\mu\text{g}$ , without using optical apparatus.

**Methods for 10-metal ions:** These were now available. Iron-ion with potassium ferrocyanide had been described by Dr. Weisz in 1954. Others now worked out were:

Ion	Reagent	Colour	Standard Solution
Fe	Ferro-cyanide ..	Blue	0.1 mg./ml.
Cu	Sulphide, Silver-nitrate	Black/Brown	0.1 mg./ml.
Ni	Dimethylglyoxime ..	Red	0.1 mg./ml.
Co	$\alpha$ -Nitro-so- $\beta$ -naphthol	Brown	0.1 mg./ml.
Al	Alizarin or Chinalizarin	Red	0.1 mg./ml.
Be	Chinalizarin ..	Violet	0.1 mg./ml.
Cd	Sulphide, silver nitrate	Blue/Black/Brown	0.1 mg./ml.
Mg	Chinalizarin ..	Blue	0.1 mg./ml.
K	Dipicrylamine or Tetraphenylborate	Red	0.3 mg./ml.
Zn	Ferricyanide + 3:3' dimethyl-naphthidine	White/Purple	0.1 mg./ml.

Fig. 1 showing seasonal variation of radio-activity in rain.

Determination of copper was best carried out as follows: The standard solution contains 0.1 mg./ml. Cu. The drops are washed into the ring zone and the filter is bathed in an acid solution of H<sub>2</sub>S, rinsed well under the tap and finally in distilled water. A yellow-brown ring of CuS appears on the paper which is not very stable. The filter is therefore bathed in a 1 per cent silver nitrate solution and washed carefully to take away excess AgNO<sub>3</sub>. A certain amount of silver sulphide is thereby precipitated on the paper in the ring which is equivalent to the amount of Cu. As silver sulphide can be formed in the same way from quite a number of metal-ions, Dr. Weisz considered that it should be possible to have only one standard scale. Methods have been worked out for 14 metal-ions using a 'universal standard scale' consisting of silver sulphide.

### Use of Silver Nitrate

Metal was washed into the ring-zone and precipitated as sulphide. This metal sulphide was bathed in AgNO<sub>3</sub> solution, transforming the metal sulphides into an equivalent amount of silver sulphide. These rings were compared with the silver sulphide 'universal standard scale.' Results were multiplied by factors which took into account the different valencies and atomic weights.

$$F = \frac{\text{atom. wgt. unknown ion}}{\text{atom. wgt. standard ion}} \times \frac{\text{valency unknown ion}}{\text{valency standard ion}}$$

Co-worker Dr. Celap had begun studies on the determination of anions using such a 'universal standard scale'.

**Determination of impurities in filter paper:** This method is considered of interest to workers using spot analysis, paper chromatography or paper electrophoresis.

Principle of the method was that soluble materials could be washed out of a circular area of the paper and concentrated in a sharply defined ring-zone. This affected concentration of the impurities as follows: the ring-oven had a borehole of 22 mm.  $\phi$ . This meant that impurities were extracted from an area of  $11^2 \times 3.14 = \text{approx. } 380 \text{ mm}^2$ . Width of the ring-zone where soluble materials collected was about 0.1 mm., circumference of the ring was about 70 mm. Consequently the area in which the soluble materials had been concentrated was approx. 7 mm<sup>2</sup>. The concentration factor was therefore  $380 : 7 = 54$ .

Selective extraction could be achieved by judicious choice of solvents—acids, ammonia, water, etc. With 0.02 NHCl (purified by isothermal distillation), for example, heavy metals were concentrated in the ring. The paper was then dried and cut in half. Iron, one of the commonest contaminants, was revealed in one half of the ring by spraying with potassium ferrocyanide. Heavy metals could be revealed in the other half by bathing in ammonium sulphide solution and washing away the excess. Finally treating the paper with silver nitrate

solution produced an equivalent ring of silver sulphide. Specific reagents could be applied to detect the presence of any particular ion.

**Ring electrography:** This had been developed by William I. Stephen and had proved useful for the rapid qualitative analysis of many ferrous and non-ferrous metals. To analyse a metal surface, on a plated article, the surface was first cleaned with a mild abrasive and a small disc of filter paper (10 mm  $\phi$  moistened with electrolyte, e.g. sodium chloride) was placed on the part to be examined. The cathode was placed on the disc and pressure applied against the spring of the plunger. The electric circuit was completed by bringing the anode (a pointed steel probe) into contact with the metal surface. A current of 10-50 milliamps was passed for 2-3 seconds and the cathode removed. The filter disc now contained cations derived from the metal specimen and was treated by ring-over procedures, i.e., it was placed in the centre of a new filter paper and was treated as if it was just a normal test drop.

Advantages of analysis by this method were its rapidity and non-destructiveness. Since only 3-5  $\mu$ g. of metal needed to be removed from an area of about 12 mm<sup>2</sup> attack on the metal surface was barely apparent. The method was very suitable for investigations on *objets d'art*, such as coins, statuettes, etc.

Mrs. Haglund, Mrs. Shalgowski, H. Shalgowski (UK AEA, Woolwich) and Dr. Haglund (Sweden)



dried and placed in contact with a film (Ilford X-ray Industrial G). Only the sectors which correspond to the ion in a active form present could give an auto-radiograph.

The possibility of obtaining a semi-quantitative conclusion was being investigated, Dr. Weiss indicated. Combination of the ring-oven technique with other analytical procedures such as polarography and chromatography have been worked out. With polarography,

the ring-oven was only used to separate the different ions.

Practical applications of the ring-oven method were in art investigations, e.g. valuable statuettes, old paintings; in controlling corrosion of different metals; in analysis of components of rubber, textiles, oils and foodstuffs after ashing organic material and extracting the ash with acid; in analysis of metals in coal ash, in the steel industry, in analysis of alloys, etc.

## Discussion on Dr. Weiss's Paper

Professor F. Feigl said that the greatest importance of the Weiss technique was in trace analysis by means of the ring-oven method. In this connection the modification just mentioned by Professor Van Nieuwenburg deserves general attention.

According to some observations it seems that iron is present in filter paper in a soluble and insoluble form. Both can be identified. With great sensitivity by spotting with a solution of Ca dipyriddy in acetic acid (formation of a red stain). The combination of direct spotting and the ring oven method may be of interest.

Professor C. J. Van Nieuwenburg (Delft, Netherlands) pointed out that the weak point of the ring-oven technique was the continuous washing. It was possible to wash into the ring as well. He could find 30 or 40 cations without any difficulty. The professor then des-

lems mentioned by Professor Van Nieuwenburg.

Dr. R. J. Magee asked whether Dr. Weiss could discuss the detection of nitrate and nitrite using the ring-oven technique. Dr. Weiss said that it was a question of reducing the nitrate to nitrite without destroying the ring formed. In a private communication from a researcher at Siemens Halske, Dr. Weiss had learned that by placing the filter paper on a zinc plate and spraying with acetic acid and solution of Griess reagent a red ring is obtained. Reduction using different metals was under investigation.

### Monsanto Phthalic Anhydride Plant on Stream at Newport

The new phthalic anhydride plant of Monsanto Chemicals Ltd. has now come into operation at Newport. This product has been produced at the company's Ruabon facilities for many years. An important variation from previous practice is said to be the use of a silicon ester, TAS 180, which is made at Newport, as a heating medium for certain parts of the process in place of high pressure steam.

### Birlec in South Africa

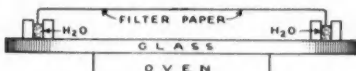
Birlec Ltd., have consolidated their entry into the South African market by opening a Johannesburg office under the management of Mr. S. G. King. The new branch will deal with project design and engineering service work in connection with melting furnaces, especially those used for making alloy steels. The address is P.O. Box 99, Witfield, Transvaal.

## Some Quantitative Analysis

Semi-quantitative analysis of certain simpler alloys had been effected using experimentally established conditions for the electrolysis and ring-colorimetry for the examination of electrochromics. Another interesting approach of combining the electrographic principle with the ring-oven method had been made by Nall and Scholey (British Admiralty Steel Laboratory, Sheffield). The method had been applied to the rapid analysis of carbon—and low alloy steels. Mn, Ni, Mo and Cr were determined.

**Autoradiography:** The ring-oven method had been applied to radioactive substances albeit on a limited scale up to the present. This method was known as autoradiography. The substances contained in a drop of test solution were concentrated on the ring oven into a very narrow ring zone. The degree of blackening of the film depended upon the activity per unit area.

Very small amounts of active substance could be identified. As an example: To a solution known to contain either labelled  $\text{SO}_4^{2-}$ ,  $\text{PO}_4^{3-}$  or  $\text{J}^-$  or combinations of these, an inactive mixture of the three ions in question as carriers was added and mixed. One drop of the resulting solution was washed into the ring-zone using 0.1 N HCl and dried. Three sectors were cut out and each was treated with a precipitating reagent for one of the ions in question.  $\text{BaCl}_2$  for  $\text{SO}_4^{2-}$ , magnesia mixture for  $\text{PO}_4^{3-}$ , silver nitrate for iodide. After thorough washing, the sectors were



The apparatus described by Professor Van Nieuwenburg

cribed a useful technique. This consisted of a glass plate on which was placed a ring reservoir of water into which the ends of filter paper were placed with the necessary ring cut out.

Dr. Weiss said that at Vienna a new apparatus had been developed, about which details would be published shortly. For that reason he had not included a note on it in his lecture. He thought that it would avoid the prob-



## APPLYING MICRO TECHNIQUES IN THE ELECTRICAL INDUSTRY

BY a number of selected examples, Mr. R. C. Chirnside (Research Laboratories, General Electric Co. Ltd., Wembley, Middlesex) illustrated the analytical and diagnostic value of some micro analytical techniques.

Analysis had a significant part to play in nuclear power for one of the major factors governing the rate of progress of nuclear power engineering was knowledge of the properties and behaviour of materials used or likely to be used. Of concern were the purity of, or impurities in, uranium; with the metal of the 'can' and its behaviour in  $\text{CO}_2$  at high temperatures; the initial purity of  $\text{CO}_2$  and any impurities appearing during use; and impurities in graphite.

Magnesium alloys were at present in use as 'canning' material and there might be future interest in beryllium and perhaps zirconium. Of importance was the oxidation resistance of magnesium and beryllium in carbon dioxide that contained small concentrations of moisture and carbon monoxide. Oxidation experiments on a wide range of magnesium alloys in  $\text{CO}_2$  and in  $\text{CO}/\text{CO}_2$  and  $\text{CO}/\text{CO}_2/\text{H}_2\text{O}$  mixtures had been carried out and the test specimens submitted to comprehensive analytical investigation.

### Test with Magnox A12

Tests on Magnox A12 had shown a significant amount of carbon deposition. A carbonate phase, detected by X-ray diffraction was confirmed chemically. The presence of magnesium fluoride was unexpected. After further experiments in which magnesium fluoride was detected again, an examination was made of the pressure vessels used for the oxidation experiments and deposits were found to consist mainly of  $\text{CaF}_2$ , a welding flux residue. Fluorine present in the 'pure' magnesium metal used to about 50 p.p.m. was determined colorimetrically on about 100 mg of metal. The colour produced with zirconium nitrate and alizarin red S was matched with prepared standards. A flame spectrometer was used to determine other elements (Na 100 p.p.m.; K 100 p.p.m.; and CaO 0.1 per cent).

The value of the conductimetric method for the determination of micro-quantities of carbon was indicated. Results were shown of carbon determinations (samples weights varied from 25 mg. to 100 mg.) on untreated zirconium metal and on several of thin layers taken successively from the surface of samples of zirconium used in oxidation experiments. Untreated material had 0.16 per cent C; 3rd layer of sample A had 0.16 per cent C; 2nd layer of sample B had 0.18 per cent C; and the 2nd layer of sample C had 0.16 per cent.

During a study of the effects on iron of  $\text{CC}/\text{CO}_2$  mixtures in different ratios, the value of X-ray diffraction as a micro-analytical technique was clearly demonstrated.

Sample	Gas	Surface Layer
2	30CO/70CO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub> strong Fe <sub>2</sub> C 2 to 3%
3	41CO/59CO <sub>2</sub>	C medium
4	51CO/49CO <sub>2</sub>	C strong Fe <sub>2</sub> O <sub>3</sub> weak

The progressive influence of the gas mixture on the surface as the proportion of CO in the atmosphere increased was clearly demonstrated by the analysis made non-destructively on the surface layer only a few hundreds of atoms thick.

A method had been worked out for the determination of cobalt in 'low cobalt' sheets. This method depended on the formation of a coloured compound of cobalt with  $\beta$ -nitroso  $\alpha$ -naphthol. This was extracted with chloroform and determined absorptiometrically. The solution of steel was treated with sodium citrate to complex the iron. Alloy steel 18/Cr/8 Ni/0.5Ti, showed 20 p.p.m. CO and Swedish iron, 30 p.p.m. CO. Samples of 200 mg. or less were used.

The boron content of graphite had to be very low because of its effect on the neutron-captive cross section. Three approaches to the problem of boron content were possible: (a) a direct spectrographic technique; (b) a combination of spectroscopy and chemistry; and (c) a wholly chemical technique. With (a) the limit varied from 0.1 p.p.m. for graphite rod samples to 1 p.p.m. for low density materials, e.g. lampblack. For (b) the sensitivity limit was independent of the original form of the sample and was 0.1 p.p.m. With (c) boron contents in the range 0.05 to 0.1 p.p.m. could be measured.

Under favourable conditions, i.e. with low 'blanks', as little as 1 mg of boron could be determined. Because of difficulties of obtaining reagents and conditions free of boron, the determinations have been carried out in a room with filtered air and with specially purified reagents. It was stressed that difficulties invariably arose from the special context in which the element occurred.

Because of the health hazard of beryllium, the maximum permissible level of atmospheric concentration of beryllium was 2.0 mg/m. metre. A batch sampling

technique is used. The test filter papers are chemically processed and 0.1 ml. (1/10th the original sample) is spectrographically examined. It has proved possible to determine  $1.5 \times 10^{-9}$  gm. beryllium on the spectrograph which in the method described means  $1.5 \times 10^{-9}$  gm. of the sample. An automatic direct reading spectrograph is now being used.

Under operating conditions for nuclear reactors, it is considered likely that  $\text{H}_2\text{O}$ ,  $\text{H}_2$ ,  $\text{N}_2$ ,  $\text{A}$ ,  $\text{O}_2$  and  $\text{CO}_2$ , and possibly other impurities are likely to be present. Analysis of  $\text{CO}_2$  and rapid determination of impurities has not been possible by conventional techniques of gas analysis. However, Mr. Chirnside reported that it was believed that they had broken new ground in the G.E.C. research laboratories in solving this problem by means of gas chromatography. Using molecular sieves as solid adsorbents and by the design and use of a highly sensitive katharometer, analysis of the gas could be carried out in a matter of minutes.

The quantity of each component determined ranged from 0.01 to  $10 \mu\text{g}$  and a sample of 10–25 c.c.s. was used in the first instance.

In a consideration of miscellaneous microchemical analysis, Mr. Chirnside dealt with some other fields of activity in the electrical industry such as determining the ratio of nickel and iron in thin metallic films polarographically; sealing alloys for glass to metal seals in vacuum devices, e.g., nickel-vobalt-iron (nickel and cobalt were determined on a 2 mg. sample by polarographic methods to about  $\pm 2$  per cent of the amount present); and evaporated films.

Summaries of some further papers and discussions will be published in "Chemical Age" next week.

### Slight Fall in Chemical Industry Capital Spending

FIGURES published by the Board of Trade show that for the first quarter of 1958 there was a drop of £2.9 million in fixed capital expenditure in the chemicals and allied trades.

		1st qtr.	2nd qtr.	3rd qtr.	4th qtr.	Year
Chemicals allied trades	1954	...	...	...	...	97.2
	1955	18.4	23.7	24.5	34.5	101.1
	1956	29.4	32.8	34.4	44.0	140.6
	1957	39.0	43.9	40.5	48.0	171.4
	1958	36.9				
All manu- facturing industry	1954	...	...	...	...	609.4
	1955	153.8	160.2	189.8	219.5	723.3
	1956	195.7	201.2	214.4	252.3	863.6
	1957	222.5	224.4	228.6	250.8	926.3
	1958	22.79				

### New Cortisone-type Hormone Developed by Olin Mathieson Division

A NEW cortisone type hormone drug has been announced by Squibb division of Olin Mathieson Chemical Corporation. Known as Triamcinolone (trade name Adcortyl), the new drug was developed to overcome the severe reactions occurring with earlier cortisone type preparations, such as an artificial sense of elation, fluid retention, increased blood pressure, stomach upsets and ulcers.

According to investigators Adcortyl can be used with greater safety in the treatment of arthritic patients. In particular, it can be used in the treatment of cardiac cases with allergies. Improvement among 86 per cent of arthritic patients has been reported and in other conditions such as bronchial asthma, allergies, and hepatitis. In a number of skin and blood disorders, a 95 per cent improvement was noted.

# LABORATORY EQUIPMENT REVIEW



## New Apparatus for Research and Industry



This annual survey of laboratory equipment features apparatus and instruments that have recently been introduced, redesigned or modified. It also includes equipment for pilot scale operations. For further details of individual items complete and return Reader Enquiry Service form on page 380.



### Two New Adelphi Products

Recent additions to the range of the *Adelphi Manufacturing Co. Ltd.*, 20 Duncan Terrace, London N1, include a hot wire ampoule cutter and a 'Junior' vacuum filling machine suitable for laboratory use.

The ampoule cutter, of the hot wire type, is mounted on a baseboard with a mains transformer giving an output of 2.5V 25W 50 cycles a.c., and the element is of bright nickel-chrome 22 s.w.g. wire mounted on two chromium plated pillars. An output of about 1,200 ampoules an hour is reasonably possible. List price is £9.

The Adelphi Junior vacuum filling machine is a simplified version of the company's standard model and has been specially designed for laboratory use. It differs from the larger machine in that any overflow passes into a standard MRC bottle, from whence it can be emptied as required, instead of passing through a reservoir and back into the bulk container. All contact parts are made of stainless steel and p.v.c. of surgical quality. Size is 9 by 5 by 17½ in., and the model weighs 10 lb. Price is £25 complete.

### Nylon Hose Couplings

The nylon free-end hose couplings produced by *Airtech Ltd.*, Haddenham, Buckinghamshire, have, in addition to industrial applications, uses in the laboratory. The moulded nylon fittings may be used to form terminals on tools, valves or supply points, or to join two hose-ends together. In each case the free, unprepared end of the hose is simply offered into the coupling or terminal, and a leak-proof joint that may be regarded as either temporary or permanent is effected by screwing tightly by hand.

### New Laboratory Equipment from Apex Construction

A traditional problem with many hand tablet presses, such as those used for the production of test samples in the laboratory, is that if they become jammed in the bottom dead centre position the punches are liable to damage in the freeing process. Now *Apex Construction Ltd.*, 15 Soho Square, London W1, in consultation with the School of Pharmacy, have produced the Apex 127 hand tablet press which transmits its punch through a ball and socket joint which is easily dismantled should the machine jam. Operating at a top pressure of 1½ tons, the machine will turn out 100 tablets per minute with diameters up to ½ in. and a maximum depth of fill of 9/16 in.

Another new Apex product is a vibratory ball mill, type 163, designed to pulverise all types of materials from 0.5 mm. feed size to 1 micron output. This operates on a new principle, for instead of the mill rotating to cause the

balls to cascade it is subjected to a mechanically-induced high frequency vibration and the balls therefore bombard the material with faster and more efficient pulverising effect. These mills are available with net working capacities from 0.36 l. up to 250 l.

The new Apex No. 200 'Mount Rapid' metallurgical mounting press is of modern

Newly developed  
'Mount Rapid' metallurgical mounting press by Apex Construction



design, having a central mould with heaters and coolers arranged so that they can be placed around this mould and removed when required. The unit is encased in a cabinet stand. A swing-away head is arranged over the mould to enable the finished specimen to be ejected. The unit is equipped with pilot lights, thermocouple type thermometer and a heat controller to control the input of current to the heaters.

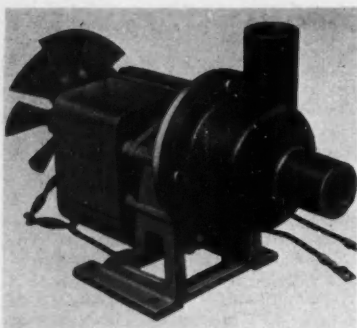
### Pressure Switch

A simple pressure switch for pressure, vacuum or liquid level control is made by *Appleby and Ireland Ltd.*, Basingstoke, Hants. Enclosed in a strong metal case with ½ in. BSP connection for pressure inlet, and grommet for electrical entry, the internal arrangement consists of a metallic capsule (beryllium copper, phosphor bronze or stainless steel) which distends proportionately to the applied pressure or head of liquid. Distention of the capsule causes the insulated platinum-iridium contact attached to the capsule mount to move towards a pre-set adjustable contact. Operating range is from 0 to 30 in. Hg. vacuum to 30 p.s.i. or a combination of both. Electrical rating is 50 volts 0.5 amps d.c.

Also new are a portable draught gauge for indicating very low pressures, a portable liquid level gauge, and a sensitive draught and low level gauge.

### New Laboratory Pump

Smallest of the range of motorised pumps made by *Appleton and Howard Ltd.*, Salisbury Street, St. Helens, Lancs, is the new laboratory-sized Minnow.

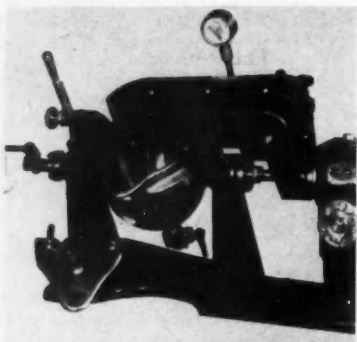


Gush general purpose Minnow pump  
by Appleton and Howard

Built for the transfer and circulation of corrosive and other liquids in experimental and pilot plants, the pump body and impeller are constructed of high density polythene with a stainless steel shaft sleeve and synthetic rubber lip seal. Capacity varies from 1 g.p.m. with a head of 10 ft., to 8 g.p.m. with a head of 1 ft.

The shaded pole motor, operating on a mains rating of 230-250 V. 50 cycles, or of 50 or 110 V 50 or 60 cycles, is fitted with a thermal overload device and cooling fan. Price is within the range £4 to £6 depending on quantity and specification.

### Steam-Jacketed Mixer



The half-gallon, laboratory size steam-jacketed mixer made by William Brierley, Collier and Hartley Ltd., Bridgefield Street, Rochdale, Lancashire. The pan is of stainless steel, with hydraulic drive to the stirrers, which continue to revolve as the pan is being tilted to empty

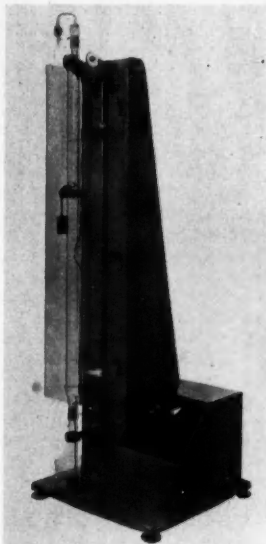
### New BTL Micro Apparatus

The new BTL silent shaker is of improved design and construction to avoid noise and vibration. Fixed traverse of 1½ in. at a rate variable up to 320 oscillations per minute enable the standard rate of 275 oscillations per minute to be obtained immediately without waiting for the apparatus to warm up. This model will hold six Kahn Racks.

Shown at the Birmingham symposium by Baird and Tatlock (London) Ltd., Freshwater Road, Chadwell Heath, Essex, was the prototype BTL micro zone-melting apparatus which can be used when only a gram or so of material is

available. The macro model, now in production, handles quantities of the order of a kilogram. Action of both models is automatic and both can be used to obtain organic compounds of something like 99.99998 per cent purity. The BTL zone-melting apparatus has been developed from the National Chemical Laboratory apparatus.

Also of interest is the company's sedimentation apparatus, ICI pattern, for the analysis of the particle size of powders



BTL zone-melting apparatus

in the sub-sieve size range. It consists of a sedimentation tube and a reservoir used for flushing samples from the bottom of the sedimentation tube, mounted in a water tank to minimise convection currents.

The prototype BTL WIRA pattern microtome knife sharpener that will sharpen all types of microtome knives is to be shown at the medical laboratory conference at Bristol University (16-18 September) with a new wide range incubator.

### High Output DC Power Source for Electrophoresis

A continuously-variable, high output power source for electrophoretic paper-chromatography, originally designed for the biochemical laboratories of a Midlands hospitals group, is now being made and marketed by Barber Medico-Electronic Laboratories Ltd., Birmingham. Operated from a.c. mains supply at 230-240 V, the d.c. output is continually variable from zero to 600 V, giving a maximum available current of 100 milliamps. Coarse voltage control is provided in ten steps, each step having a continuous fine control over its entire range.

### Stainless Steel Filters

Carlson Pilot-Princess filters, employing ready-made filter sheets of asbestos and cellulose, are made for laboratory purposes with filter surfaces as small as 3 sq. in. Manufactured in stainless steel

by John C. Carlson Ltd., Newman Street, Ashton-under-Lyne, Lancs, the apparatus is designed for filtering such liquids as penicillin, streptomycin, insulin, sera, liver extracts, etc., the output varying according to the number of filter plates used and the nature of the liquids concerned.

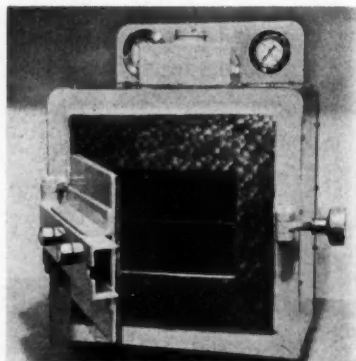
### Two-way Fan to Reduce Dust Infiltration

The Colt two-way mains-operated fan, manufactured by Colt Ventilation Ltd., Surbiton, Surrey, is an effective means of providing general ventilation in laboratories. By continuously replacing the extracted fume-laden, vitiated air by the same volume of fresh air from outside, the pressure in the laboratory is maintained and dust entry by infiltration is minimised. The fan may be fitted with filters and a heater if necessary.

### Vacuum Shelf Drier

Designed primarily as a laboratory machine for carrying out drying tests to be later reproduced in C-3 production vacuum shelf driers, the Calmic C-3 laboratory vacuum drier, made by Calmic Engineering Co. Ltd., Crewe, Cheshire, has proved so popular for general purpose laboratory use that it is now being marketed as such. The drying compartment is 11 in. deep from back to front, 8 in. wide and 6 in. high, and is arranged with two shelves 11 by 8 in., giving a total tray area of 1½ sq. ft. It is available with two temperature ranges—20° to 90° C and 80° to 150° C.

In addition to the thermostatically-controlled electric immersion heater in the oil which circulates around the com-



New Calmic laboratory vacuum shelf drier

partment, there is an overriding safety thermostatic controller. If a temperature of 130° C is exceeded, the electric heater is automatically cut out and does not cut in again until manually reset—a feature of value when the laboratory drier is required to work overnight unattended.

### 'Build-as-you-Go' Polarograph

A new polarograph made by Cambridge Instrument Co. Ltd., 13 Grosvenor Place, London SW1, combines advantages of several previous types, can be purchased in a simple and inexpensive form, and by adding auxiliary apparatus (some of which the user may already possess)

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**New Cambridge general purpose polarograph**

becomes a rapid and accurate pen-writing instrument with facilities similar to those of the Cambridge direct-writing polarograph. All standard accessories can be used with the new general purpose polarograph including the Cambridge Univector unit, and it is suitable for use with dropping mercury or other electrode systems.

The basic instrument has adjustments for slide-wire current standardisation, voltage range, sensitivity, zero setting and damping and is contained in one small case. A specially-designed electrode stand is available, but where economy is more important than convenience, a suitably arranged retort stand will serve.

In its simplest form, the polarograph is used with any galvanometer of suitable specification and the current/voltage curve is plotted manually. By the addition of a suitable potentiometric recorder, it becomes a direct-writing instrument, and draws the polarograms automatically. Sensitivity is variable in ten related steps, each value being about  $1\frac{1}{2}$  times the preceding, covering a total range of 1/1 to 1/50.

### Ultrasonic Cleaner

An ultrasonic power generator suitable for the rapid cleaning of delicate pieces of laboratory equipment is made by *Dawe Instruments Ltd.*, 99 Uxbridge Road, London W5. Listed as the 250 w Soniclear type 1156, the instrument consists basically of a valve oscillator and a transducer. The mains-powered generator supplies high frequency electrical impulses at around 40 kc/s to the transducer, which is made from barium titanate. Transducers can be operated with liquid temperatures of up to 160°F, although higher temperature applications may be carried out by keeping the hot solution in a separate inner container and transmitting the ultrasonic energy through a water bath fitted with cooling coils.

### High-stability Graticules

A successful result of sponsored research by Scientific Instrument Research Association on behalf of *Graticules Ltd.*, 57/60 Holborn Viaduct, London EC1, is the commercial production of a range of highly stable graticules under the trade name 'maXta'. The production process is a technique combining a photographic and a physical chemical process which fixes the photographic image into the surface of the glass. As a result, maXta type A graticules, for example,

will resist all abrasion not actually harmful to the glass itself; and since maXta-surface imaged graticules are resistant to heat, light, fungal and most other forms of chemical attack, they can be used in instruments, and in places, where it has hitherto been impossible to use graticules at all.

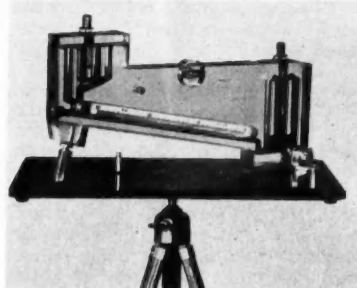
The maXta process gives a sharply-defined image with lines down to three microns thick, opaque to all forms of light and particularly suitable for transparent grey images, giving sharp definition at the edges and good transparency both in clear and grey areas.

### Davisil Glassware

The new illustrated catalogue of *Davey and Moore Ltd.*, Brimsdown, Middx, covers their range of Davisil borosilicate glass and flint glass laboratory and chemical apparatus. The narrow mouth standard stopper for reagent bottles is now dustproof up to 1,250 ml. New additions include vacuum desiccators, Dreschel bottles, vacuum type bell jars, and a Flexicon aspirator.

### Micro-manometer in Transparent Perspex

The single tube pressure gauge constructed in a block of transparent plastics by *Combustion Instruments Ltd.*, 61 Belsize Lane, London NW3, is claimed to be virtually unbreakable and unspill-



**Combustion Instruments' Mk 4(a) micro-manometer**

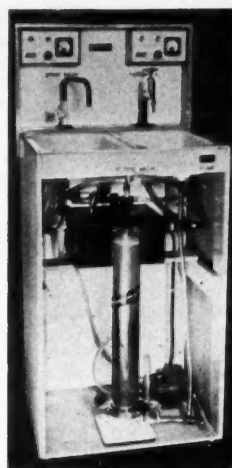
ble. The micro-manometer illustrated, Mark 4a, is an inclined tube gauge graduated, when oil filled, from 0 to 1.20 in. w.g. in hundredths, and from 0 to 1.50 in. w.g. when water filled. This model costs £21 10s. with panel mounting. The Mark 1 range, said to be accurate to 0.025 in. w.g., is rated from  $4\frac{1}{2}$  in. w.g. (model 1a at £4 10s.) up to 40 in. w.g. (model 1i at £30).

### Elga Recirculator

Designed specifically for the provision of solid-free water for washing of slides and laboratory glassware is the Elga Recirculator, produced by *Elga Products Ltd.*, Railway Place, London SW19.

The Recirculator permits air or oven drying. No stains or deposits will remain, it is claimed. As the used washing water is not run to waste but recirculated through the column containing ion-exchange resin, effluent costs are reported to be negligible. Column life has been found to be long and weeks of washing may be carried out between regenerations.

**Elga Recirculator**



The unit illustrated is not a standard item. Sinks can be adapted to suit individual requirements. The ion-exchange resin column is contained in the centre of the cabinet. A water-storage tank is contained behind the column.

Washing may be carried out in two ways, i.e., by spray washing or by flooding from the base of the sink. When the resistivity of the influent water equals that of the effluent, washing is considered complete. A meter indicates effluent purity and another, effluent resistance.

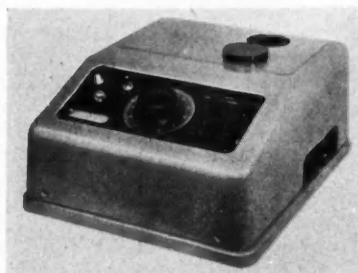
Price of the unit is approximately £165, according to design.

### EEL Fluorimeter

A compact self-contained instrument designed to measure the fluorescence of liquids is the 'EEL' Fluorimeter introduced by *Evans Electroscelenium Ltd.*, Bishop's Stortford, Herts.

Light from a high pressure mercury lamp passes through glass primary filters, which isolate the desired region of the ultra-violet, to irradiate any one of the three cuvettes carried in a rotating sample holder. The fluorescent light emitted by the solution passes through interchangeable secondary filters to fall on a high sensitivity photo-multiplier tube.

The fluorescent light emitted by a reference solution passes through similar secondary filters to fall on a second multiplier tube. The two photo-multipliers are connected in opposition to a robust, built-in, null-point galvanometer which is brought to zero by means of a high precision potentiometer.



**EEL fluorimeter**

The standard unit is designed to take selected test tubes of 4 ml. capacity. The unit is removable and may be replaced by a holder designed to take either tubes of small capacity or rectangular cells if required.

The primary filters, which are interchangeable, are of glass and comprise filters OX1 and OX2 which irradiate the sample primarily at wavelengths of 365 m $\mu$  and 436 m $\mu$  respectively.

The secondary filters are also interchangeable and any of a set of eight spectrum filters can be provided, having peak transmission from 450 m $\mu$  to 670 m $\mu$ .

Sensitivity of the instrument is stated to be of a high order, for example the full range of the calibrated dial may be employed with a standard solution of quinine sulphate at a concentration of 0.01  $\mu$ g. quinine/ml.

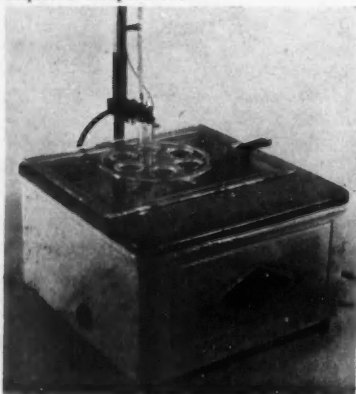
### Tinsley Polarographs and Thermostat Bath

Two polarographs, the Mark 19 recording instrument and the Mark 16 polarograph minor, together with electrolysis stands and ancillary equipment are available from *Evershed and Vignoles Ltd.*, Acton Lane Works, Chiswick, London W4.

The Mark 19 is an all mains instrument, the polarising potential being supplied from a stabilised rectified power unit. The derivative circuit is operated by a simple switch change over. The coarse and fine zero controls enable the operator to read a small step following a large one, and the counter current control permits the cancelling out of non-faradaic and a large residual current at high sensitivities.

The manually operated Mark 16, designed specifically for the smaller laboratory and the teaching institute, has a linear 9 in. scale and although non-recording incorporates a derivative circuit. Maximum sensitivity is 0.02 microamps for full scale deflection.

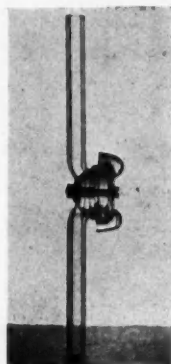
The thermostat bath for use with Tinsley polarographs has been modified. The lid is of Perspex and will be unaffected by mercury and most dilute solutions encountered in polarography. The centre disc which holds six cells, may be rotated beneath the dropping electrode so that six solutions can be polarographed consecutively after reaching the required temperature.



Tintometer comparator discs

Interior of the bath is of stainless steel and the outer case of heavy gauge aluminium finished in cream stove enamel. Specially designed removable heaters and baffle enable the temperature to be kept within  $\pm 0.25^\circ\text{C}$ . There is a  $40^\circ\text{C}$  range of temperature control from any pre-set origin, with a maximum temperature of  $100^\circ\text{C}$ .

### Stopcocks for Micro Apparatus by W. G. Flaig



Stated to be suitable for all micro-chemical apparatus and burettes is this new addition to Exelo interchangeable stopcocks marketed by W. G. Flaig and Sons Ltd., 39 Waterloo Road, London NW2. This is a 2 mm. bore size and is available in both borosilicate and soda glass with ordinary arms or burette type

### Baker Deoxo Purifiers and Platinum Crucibles

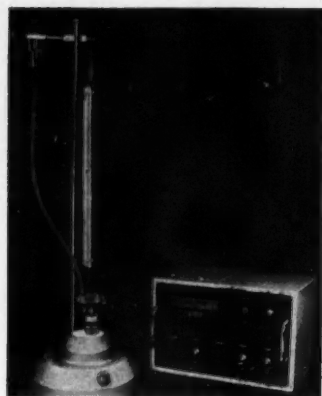
The recently introduced Polygon range of platinum crucibles was shown at the microchemistry symposium by the Baker Platinum Division of *Engelhard Industries Ltd.*, 52 High Holborn, London WC1. Features claimed are ease of pouring and handling. Also on view was a selection of the standard range of platinum laboratory ware; and small laboratory models of the Baker Deoxo purifiers, which provide for the catalytic hydrogenation or oxidation of certain gases. By this process gaseous impurities, such as oxygen, hydrogen and carbon monoxide can be removed with a remaining impurity of less than one part per million.

The Hersch oxygen meter, designed to measure traces of molecular oxygen in other gases, such as nitrogen, hydrogen, argon, helium, ethylene, etc., in the range 0-10, and 0-100 per million, was also featured.

### Potentiometric Microtitration Apparatus

A new apparatus for the accurate determination of chloride in small quantities of blood plasma, serum, urine and cerebro-spinal fluid has been developed by *A. Gallenkamp and Co. Ltd.* in co-operation with Dr. I. D. P. Wootton of London Postgraduate Medical School.

It is possible to determine quantities of the order of 4 m.eq./litre, that is, the chloride content of the normal person's plasma diluted 25 times, Gallenkamp's claim. The apparatus is accurate to better than  $\pm 1$  per cent of the total quantity determined when used by an experienced operator. Each determination takes about two minutes and it is possible to make a hundred or more determinations a day.



Gallenkamp's new potentiometric micro-titration apparatus

Chloride in 5 or 10 ml. samples is precipitated by silver nitrate solution run from an accurately calibrated 5 ml. burette. The end point is indicated when the galvanometer spot reaches a predetermined reading. This reading is obtained by periodic checks against standard chloride solution, and is indicated when the galvanometer spot comes to rest.

The apparatus is in two parts: a titration stand and an electronic potentiometer. The burette, which has a built-in platinum-silver electrode system, is carried by a vertically sliding carrier. The self-filling burette is supplied from a reservoir attached to the support rod. Sample and titrant are continuously mixed by a constant speed magnetic stirrer built into the stand.

The Gallenkamp semi-micro centrifuge is low priced and is designed for short period sedimentations to replace slower and more cumbersome methods of filtration. Speed is approximately 2,000 rev./min.

### Semi-Muffle Kilns

A range of semi-muffle laboratory kilns designed to meet the exacting requirements of experimental duties for temperatures up to  $1,530^\circ\text{C}$  are produced by the Allied Engineering Division of *Ferro Enamels Ltd.*, Wombourne, Wolverhampton. Muffle design and element displacement are so engineered as to ensure uniformity of temperature in the firing chamber and even temperature increase throughout the heating cycle.

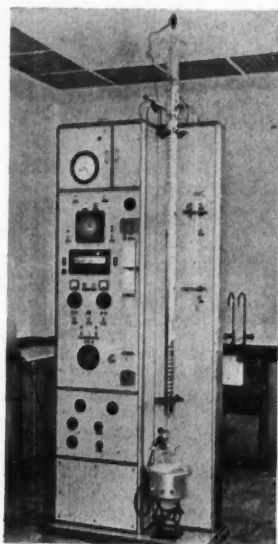
Sizes range from the LK 1, rated at 9 kW with a chamber size of 9 by 9 by 9 in., up to the LK 6, of 24 kW and chamber size 14 by 20 by 14 in. Control is effected through voltage regulating type air-cooled protected transformers equipped with fine and coarse six point switches, giving 36 steps of control over the complete voltage range. Mercury type relays are standard.

### Laboratory Fractionating Column

The free-standing self-contained precision fractionation apparatus for laboratory use, manufactured by *Glass Developments Ltd.*, Sudbourne Road, Brixton Hill, London SW2, was designed in the BP research laboratories for the separa-

tion and analysis of complex petroleum hydrocarbon mixtures, but it is suitable for general use with all volatile organic materials. Distillation under vacuum and automatic take-off operation could be undertaken.

The column has an overall length of 1,830 mm., a packed length of 1,665 mm., and an internal diameter of 21 mm. A silvered vacuum jacket is fitted throughout its length to minimise heat loss. To enable operation at column head temperatures up to 200°C expansion bellows are fitted in the outer jacket wall to 'take up' differential thermal expansion between the inner and outer jacket wall. A number of packing materials can be used in the column. Among the most efficient of these are 1/16 in. Dixon gauge rings which, when installed, give



Glass Development's new fractionating column

the column after flooding 107 theoretical plates. Attached to the column is a reflux ratemeter. Measurement is performed by placing a steel ball in the hole in the platform surrounding the vapour upriser, and noting the time required to fill the graduated annulus around the vapour tube by the liquid flowing down the column.

### 1/4 Second Electronic Recorder

Honeywell Controls Ltd., Ruislip Road East, Greenford, Middlesex, have an electronic recorder described as the fastest-acting large-chart recorder available; it traverses the full 11 in. chart width in a quarter of a second. Its split-second response and high accuracy is stated to be finding important applications in guided missile development, work on analogue computers, spectrographic analysis, and wherever permanent records of fast-changing variables are required.

Special features in line with its high speed of operation include continuous standardising, eliminating the interruptions of periodic standardising, card-mounted resistors for quick range-chang-

ing, an improved pen assembly and a new 'plug-in' amplifier unit eight times as powerful as that used in slower instruments. Dead spot is less than 0.1 per cent, and calibrated accuracy  $\frac{1}{4}$  per cent of full scale.

### Speedomax H Recorder

The one-second Speedomax H recorder, developed by Integra, Leeds and Northrup Ltd., Edgbaston House, 183 Broad Street, Birmingham 15, is proving of value in association with gas chromatography equipment and other such analytical processes where a high speed, low range recorder is desirable.

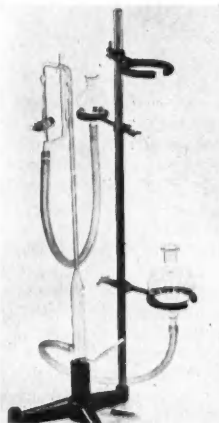
The type H indicator has a 23 in. circular scale around which a large black pointer moves to indicate value of the measured quantity. Speedomax H instruments use a mill-balance measuring circuit with a triple-filtered amplifier, a slide wire and automatic standardisation (for potentiometer measuring circuits). Accuracy rating is  $\pm 0.5$  per cent of range span; span step response time rating is 5 seconds (1 second rating also, for thermocouple and multivolt ranges only).

### Griffin Mercury Seal Nitrometer

In the improved micro-nitrometer\* marketed by Griffin and George (Sales) Ltd., Ealing Road, Wembley, Middlesex, the use of a mercury seal obviates the contact of tap grease and potassium hydroxide solution which has been found to be a weakness of conventional forms of the apparatus. Unobserved leaks and the need for frequent tap cleaning are thus avoided.

The absorption vessel, with a side-arm for the alkali levelling tube and another with inlet-gas bubbler, has a graduated upper stem with a capillary tube fused to its end. This tube leads horizontally, then downwards, to a capillary tap with a fiduciary mark in the length above the tap. The lower side of the tap is fused as illustrated into a wider-bore tube, with a side-arm curved to stand vertically and having its upper end open to air. A rubber tube connects the lower end to a reservoir for mercury.

Between determinations the capillary remains filled with nitrogen saturated



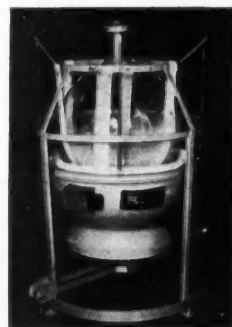
New Griffin and George mercury seal nitrometer

with water-vapour in equilibrium with the alkali solution. At the start of a determination the mercury is brought up to the fiduciary mark and the tap closed. When satisfactory micro-bubbles are obtained in the solution, the level of the alkali reservoir is changed to make the liquid level in the nitrometer and reservoir equal, and the reading on the nitrometer is recorded (e.g., 0.1 to 0.15 ml); after the combustion and sweeping stages, the reading is again recorded and the volume of nitrogen calculated in the usual way. The level of the mercury at the fiduciary mark is checked (this has never been observed to alter).

\* Cropper, F. R., *Analyst*, 1954, 79, 178.

### Special Isomantles

Special Isomantles are available from Isopad Ltd., Barnet-by-Pass, Boreham Wood, Herts. The latest version com-



Isomantle support type EXO for exothermal reactions

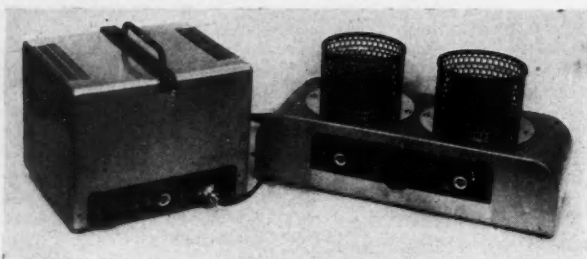
prises a heating mantle for round or pear-shaped bulbs 12 to 25 ml. with variable control. To cater for individual requirements special designs are made by the company. There is thus an Isomantle unit heating four metal beakers with stirrers mounted on supporting rods. Isomantle support type EXO is provided for those chemical processes that require the quick removal of the heating mantle from the flask in order to obtain rapid cooling. The Isomantle is spring-mounted and can be lowered away by aid of the lever whilst the flask is held by a glass fibre harness.

### Laboratory Ultrasonic Cleaning Equipment

Sole distributors of the Mullard 20 kc and 40 kc laboratory ultrasonic cleaning equipment are Kerry's (Great Britain) Ltd., Warton Road, Stratford, London E15.

The Mullard 20 kc radial bench cleaner has a laminated nickel transducer which focuses ultrasonic energy, giving increased cavitation for removal of difficult contamination. Even microscopic particles of dirt, it is claimed, are removed efficiently leaving the surface ultra-clean easily and quickly. The equipment is arranged for bench mounting and the glass beaker permits any type of cleaning agent to be used, the beaker being easily removed for cleaning. The price of the generator (type L277) and cleaning bath (L276) is £197.





**Kemp's Mullard 40kc. bench type ultrasonic cleaning equipment**

The Mullard 40kc pulsed double beaker cleaner permits two distinct cleaning operations or a pre-wash and final rinse for heavily contaminated articles. Price: twin beaker is £237 and one beaker £197.

### Pyrex Range of Microchemical Ware

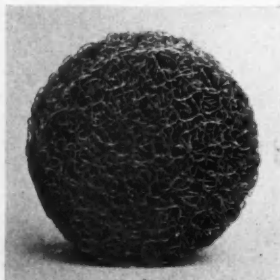
The new range of micro and semi-micro apparatus in Pyrex developed by *James A. Jobling and Co. Ltd.*, Wear Glass Works, Sunderland, includes beakers, ranging from 5 ml. to 30 ml.; conical flasks, from 5 to 25 ml.; funnels; boiling flasks, from 5 to 25 ml., with either conical or round bottoms; distillation flasks in 10 and 25 ml.; Kjeldahl flasks in 10 and 30 ml.; semi-micro flasks both pear-shaped and round bottomed, semi-micro distillation flasks; pipettes in 1 and 2 ml.

Other items in the range include a combined reflux and distillation apparatus with 1,800 mm. long vertical condenser and 95 mm. long side condenser; Pozzi-Escot steam distillation apparatus with inner vessels; 'cold finger' reflux apparatus; Willstatter 'nail' apparatus; Pregl filter with crystallising tube; conical centrifuge tubes; and distillation under reduced pressure apparatus, in both 15 and 25 ml.

### New Packing Material for Super Fractionation

New packing material for small and medium-sized columns for super fractionation is KnitMesh Multifil produced by *KnitMesh Ltd.*, 36 Victoria Street, London SW1. Distributors are Weinreb and Randall Ltd., Greenside Road, Croydon. The new material is available in copper, Monel and stainless steel.

It is knitted from a yarn comprised of a multiplicity of filaments that has capillary characteristics which 'hold' the liquid by surface tension, in a similar manner to that of fine wire mesh, and



**New KnitMesh Multifil distillation material**

this together with a surface area of about 600 sq. ft./cu. ft. is stated to account for the much higher efficiency for distillation purposes, than can be achieved by a simple filament material. It is said to be particularly competitive in price and with 95 per cent free gas space, it is claimed to have excellent throughput characteristics with very low pressure drop per theoretical plate. (Generally less than  $\frac{1}{4}$  in. w.g. per equivalent plate over the normal working range.)

Multifil is knitted in tubular form, which is crimped or ridged diagonally or in herringbone form. The flattened tube is then folded back on itself once, so that the ridges in contact with one another are approximately at right angles. The once folded material is then rolled up and the column filled with the necessary number of rolls to make up the desired packed height.

The vapour load factor  $V_s \sqrt{\rho_v}$  (where  $V_s$  = superficial vapour velocity ft./sec. and  $\rho_v$  = mean vapour density (lb./cu. ft.) takes care of different vapour densities but allowance should be made for wide variations. HETP values in the region of 1 m. to 2 m. are stated to be realisable for small columns but this efficiency deteriorates with increasing elements.

The manufacturers believe that the relatively low price of Multifil may make it an economic choice even for large size columns.

### New Humidity Controller

Two new instruments have just been released by *A. M. Lock and Co. Ltd.*, Prudential Buildings, 79 Union Street, Oldham, Lancs. These are the Lock thermo-switch and the Lock humidity controller.

The electronic thermo-switch, designed for controlling temperature, includes these features: long term calibration accuracy; on/off control action on a temperature differential of  $\pm 0.1$  per cent of range coverage; available for use in temperature range  $-200^\circ\text{C}$  to  $+500^\circ\text{C}$ ; uses interchangeable resistance sensing elements.

The switch is designed to maintain temperatures at a pre-set value, and provides immediate relay action for any tendency to deviate from it. Mains supply requirements are 200/250 volt 50 c/s for standard equipment.

The Lock humidity controller uses a new type of electrical sensing element which, the manufacturers claim, opens up many new fields of application. It provides a simple, reliable humidity

controller of long term calibration accuracy. The instrument will provide a warning of deviation from a required humidity, or in those applications for which on/off control is satisfactory, will control the humidity at a pre-set level. It is capable of detecting changes of the order of  $\pm 2$  per cent.

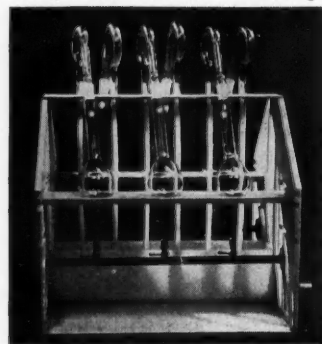
Two ranges are provided, selected by means of a simple toggle switch. High range is 25 to 100 per cent humidity and low range is 5 to 55 per cent humidity.

The sensing element is manufactured from a special plastics material which is suitable for use in a wide range of industrial conditions, not previously suitable for humidity sensing elements. It can be used in temperatures up to  $180^\circ\text{F}$ .

### Nitrogen Digestion Apparatus

Designed to carry out multiple Kjeldahl digestions on the bench is the NDA nitrogen digestion apparatus developed by *Loughborough Glass Co. Ltd.*, Loughborough, Leics. It occupies very little space. No fume cupboard is necessary; connections to gas, water and waste only are required.

The apparatus consists of a stand of an asbestos-cement heat-resistant composition, which will accommodate up to



**Nitrogen digestion apparatus by the Loughborough Glass Co.**

six Kjeldahl flasks of 200, 300, 500, 800 or 1,000 ml. capacity by means of adjustable shelves and burners. For removal of acid fumes a multiple water spray unit constructed in polythene is provided.

Semi-micro apparatus has been specially designed by this company for teaching organic chemistry at all levels. In the glassware B14 joints are used throughout and the flask sizes are 25 ml. and 50 ml. capacity. The traverse on the jaws of the clamps is limited to accommodate tubing up to 18 mm. O.D. maximum and their closing is spring controlled.

In association with *British Filters Ltd.*, Loughborough have developed the Loughborough water-deioniser. It will deliver deionised water at a pressure only a few pounds below mains pressure. A plastics-coated metal cartridge contains ion-exchange resins and to avoid any tendency for these resins to separate by flotation, the unit is designed for downward flow. Water of superior quality to normal distilled water as defined by BP1953 is produced, state the manufacturers. It is not necessarily pyrogen-free.

This deioniser will produce 50 gall. of deionised water from water containing 230 p.p.m. ionisable solids. Price of the deioniser complete is £45; spare cartridge £18, cartridge exchange service £1 5s.

### Mass Spectrometers for Isotopic Analysis

The mass spectrometer in the MS2 series produced by *Metropolitan-Vickers Electrical Co., Ltd.*, Trafford Park, Manchester 17, is arranged for isotopic analysis of solids and is designated the MS2-S. Where a laboratory requires an instrument for general isotopic analysis, a gas source of the electron bombardment type can be supplied in addition to the thermal ionisation source. The stabilised supplies for the different types of source must also be interchanged. Such an instrument with facilities for either solid or gas sources and fitted with a gas handling system of the double inlet type is known as the MS2-SG.

The MS2-S and MS2-SG spectrometers comprise three units; a tube unit, a control unit and recorder unit. These are separate cubicles with electrical interconnection only.

In the specification of performance claimed for the instruments the range of mass covered is mass 2 to 450 with full accelerating voltage. Based on a 1 per cent valley between equal peaks one mass unit apart, the resolution varies between 150 and 225 depending on sample technique. With natural uranium, the valley between  $U^{235}$  and  $U^{238}$  is less than 3 per cent of the small peak ( $U^{235}$ ) or approximately 0.02 per cent of the large peak ( $U^{238}$ ).

Samples down to .01 cc. can be measured if the reservoir volume is reduced.

### MSE Highspeed 17 Centrifuges

Two versions are available of the new MSE high speed 17 centrifuge—with refrigeration and without—which is made by *Measuring and Scientific Equipment Ltd.*, Spenser Street, London SW1.

The main feature of these two new models is the combination of very large capacity with high speeds and are said to meet present needs in laboratory centrifuging. Capacity and speed specification, available ranges from 6 by 250 ml. angle head (10,000 r.p.m.) to 16 by 15 ml. angle head (17,000 r.p.m.). There is also an 8 x 5 ml. enclosed swing-out head (15,500 r.p.m.).

All the heads are fully sealed and can be taken off the machine spindle without removing the head lid. The smaller tubes are provided with individual sealing arrangements. Adaptors are available to accommodate smaller tubes (25 and 7 ml.) in the angle heads.

Both models are provided with a number of control refinements such as push button speed control for any speed between 0 and 17,000 r.p.m.

An air circulation system is used for the model without refrigeration. Air is drawn into the centrifuge through an opening at the back of the lid, passes over the rotating head and is finally expelled at the bottom of the centrifuge

cabinet. This ensure minimum temperature rise even during high speed centrifuging.

### Redesigned Water Stills

Vitreous and chrome finishes have now been given to the Manesty 'OB' and 'OOB' automatic water stills by *Manesty Machines Ltd.*, Speke, Liverpool 19. These strongly constructed stills have a boiling chamber of heavy iron casting, the condenser pipe of steel, the lid and baffle cup of toughened glass with inner baffle of stainless steel. The condenser tube is now of stainless steel and terminates in a swaged down nozzle thus obviating the use of a separate nozzle and attendant washers. The weir chamber has been redesigned and is fitted with a removable plug. All cast iron parts are vitreous enamelled as is the condenser. Other parts are chromium plated.

Output of distilled water produced by the 'OOB' still is six to eight pints per



Manesty OB automatic water-still

hour, and of the 'OB' still three to four pints per hour. Quality of the distilled water is stated to be above the BP requirements.

### Marconi Fluoroscopic and Radiographic Cabinet

A new fluoroscopic and radiographic cabinet is available from *Marconi Instruments Ltd.*, St. Albans, Herts. The basic design of this unit provides for the direct visual inspection of small parts by X-ray fluoroscopy. A 5 in. deep inspection drawer, mounted immediately above the tubehead, will accommodate objects up to 12 in. by 10 in. Facilities for removal of the tubehead make it possible to carry out external radiography of objects which are too large to fit the inspection drawer. The unit is completely self-contained and, when fitted with castors, it can be moved from one site to another with the minimum of difficulty.

Among the applications for equipment of this type are the examination of plastics and hard-rubber mouldings of various kinds with and without inserts of different materials; checking for incorrect filling, internal cracks, and plasticity of opaque materials such as toothpaste (measured from the position of a steel ball sinking through the specimen).

The sheet steel cabinet is lead-lined where necessary for safety and contains



Marconi fluoroscopic and radiographic cabinet, type TF1601

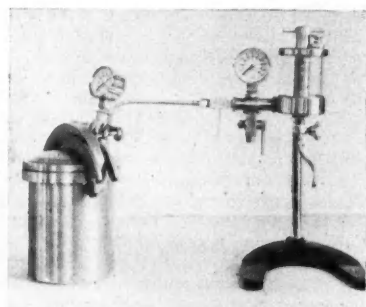
the complete equipment. A sloping front panel, mounted to the right of the fluoroscopic viewing hood, has all the relevant controls, meters and indicators mounted on it.

### Laboratory Metafilter

Where it is desired to carry out tests on a small scale in order to ascertain the suitability of Metafilters for any purpose, *Metafiltration Co. Ltd.*, Belgrave Road, Hounslow, Middx, have available the laboratory Metafilter. This consists of a small ring-type model of one twenty-fourth of a square foot filtering area, contained in a glass vessel so that the operations of bed formation and filtering can be viewed. The liquid to be filtered is forced by air pressure into the bottom of the glass container, the filtering bed having previously been formed on the surface of the filter pack or column. Alternatively the filter bed may be formed by mixing the filtering medium with liquid to be filtered and running sufficient through to ensure a clear filtrate before collecting the main bulk. A delivery pipe at the base takes away the filtrate. A pressure gauge and petcock are provided.

The filter is claimed to be convenient for dealing with small quantities of liquid, which can be filtered using pressures of up to 100 p.s.i. Used under vacuum, the pack from the glass container is removed.

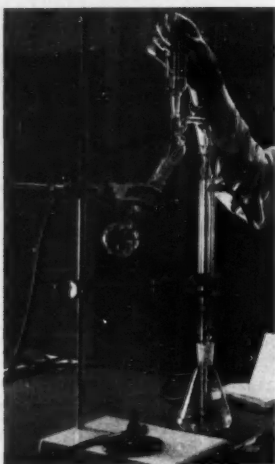
The motorised laboratory filter pro-



Laboratory Metafilter

duced by Metafiltration Co. is stated to have all the advantages of a pumping system with a small filter made of 18/8/MO stainless steel and body of highly resistant glass.

### Kjedahl Apparatus



Kjedahl distillation equipment supplied by Quickfit and Quartz, Ltd., affords a convenient means of determining nitrogen content, especially of naturally occurring compounds. The semi-micro apparatus shown here is available in either 50 ml or 100 ml capacities. Steam is generated separately and passed into the flask and distillation can be completed in 15 minutes. The same flask can be used for both distillation and digestion

### High Speed Infra-Red Spectroscopy

A new type of photoconductive cell designed for high-speed infra-red spectroscopy, including the analysis of gases, has been introduced by Mullard. The cell (type ORP10) has an uncooled indium antimonide element and is sensitive to infra-red radiations of wavelengths up to 8 microns.

The indium antimonide cell has a wider response range in the infra-red than any other type of detector operating at room temperatures, with the exception of thermal devices such as the bolometer and thermopile. These, however, are too slow in response to be suitable for high-speed spectroscopy, whereas the response time of the indium antimonide cell is extremely short. The optimum scanning frequency to give satisfactory recording on a cathode ray tube, and to achieve simplicity in the design of the cell amplifier, is between 1 kc/s and 10kc/s and frequencies of this order are easily within the scope of indium antimonide.

The construction of the cell is essentially simple, consisting only of the photoconductive element enclosed in a flat protective case, with two flying leads for connection. The sensitive area is a narrow rectangular strip measuring

6.0mm. by 0.5 mm. and the overall dimensions are 12mm. by 17mm. by 1.5 mm. thick. It is believed that the ORP10 may influence the design of future instruments since it not only enables the comparatively expensive exit slit to be dispensed with, but can readily be mounted in banks or stacks.

Of the 37 chemical groups with fundamental absorption spectra in the infra-red no fewer than 34 can be studied with the cell. The main atmospheric absorption bands also fall within its range.

### Multiple Titrations Dispenser

An automatic dispenser for multiple titrations to accelerate and facilitate the normal serological and other testing methods which involve repetitive titrations has been designed by Nash and Thompson Ltd., Oakcroft Road, Chesham, Surrey.

Special racks which carry a row of ungraduated pipettes are set on the lifting platform by means of which the 12 pipettes can be inserted simultaneously into position in the head of the apparatus. In this position the pipettes are individually connected with 12 pumping units. The test tube racks containing 12 test tubes or a common trough can be brought to the open tips of the pipettes by the lifting platform and measured volumes of fluid are aspirated into each pipette. The whole, or portions of, the contents of the pipettes can then be expelled into rows of test tubes by the reverse movement of the volume control mechanism.

### Ultra Micro Balance

For weighing with an accuracy of 0.1 microgramme and capable of handling loads of up to about 0.5 gm. there is the quartz fibre ultra micro balance recently introduced by L. Oertling Ltd., Cray Valley Works, St. Mary Cray, Orpington, Kent. The instrument is stated to be 10 times more sensitive than the conventional micro balance. (See also CHEMICAL AGE, 22 March 1958, p. 554.)

The firm's range of two pan balances, first introduced in 1957, has now been extended to include a semi micro balance with a sensitivity of 0.1 mg. per scale division (model 12FO7), and with automatic weight manipulation up to 1 g. The full range now comprises 14 models, with different sensitivities, each designed for maximum flexibility, reliability and trouble-free use.

### One-Pan Balance

Optical-Mechanical (Instruments) Ltd., 17 Station Road, Egham, Surrey, have recently announced a new one-pan balance of the Mikrowa (Swiss) series of high capacity, known as FW/54/4. This instrument is identical in appearance to the AW/10 Ultra-Speed balance but has chromium steel pans of 6 in. diameter, the height of the stirrup being 10 in. for large samples.

As is normal practice, it is equipped with an optical scale of approximately 100 in. in length reading to 1/10,000 part

of a total scale range by Vernier which in this case is 0-10 gm. by 1 mg. steps.

Built into the instrument are two control knobs, while a counter for automatic weight application built into the rear compartment of the instrument will give a total capacity of 600 gm. The balance is oil damped whereas the more sensitive AW/10 Ultra-Speed is air damped and extremely high speeds are possible. The weighing time is in the order of 6-8 seconds.

Future developments by the company in the field of One-Pan balances are the AW/50 Exactor and the AW/100 Semi-Mikro both of which have a maximum load of 100 gm. to an accuracy of .02 and .01 mg. respectively.

### Versatile 'Wet' Gas Meter

Latest in the range of integrating gas meters of both 'wet' and 'dry' type made by Parkinson Cowan Industrial Products, Cottage Lane City Road, London EC1, for measuring almost any gas on a laboratory scale is the E40. This is a wet meter of 1/10th cu. ft. per revolution, 40 cu. ft. per hour, or alternatively 2½ litres per revolution, 1,000 litres per hour. Accuracy is  $\pm 0.5$  per cent over the whole range.

### New Paterson Instruments

Two new water testing instruments recently developed by the Paterson Engineering Co. Ltd., 129 Kingsway, London WC2, are a turbidity meter and a laboratory flocculator. The turbidity meter is designed to obtain the precise accuracy and reliability of an instrument of the nephelometer type, while reducing to a minimum the cost of the equipment and the skill needed to obtain accurate determinations.

To obtain a reading of turbidity it is only necessary to turn the indicating knob until the slider touches but does not obscure the last visible spot. An arrow on the indicating knob gives, on a circular scale, a direct reading in units of turbidity, i.e. milligrams of silica per litre. The readings on the scale are standardised before calibration against finely divided silica suspended in doubly distilled water, a turbidity of one unit representing about 100 million particles of a diameter of 0.00001 cm. or 0.000004 inch per millilitre of silica suspension.

Normal motor output of the Paterson laboratory flocculator is 45 r.p.m., giving a speed range on the flocculator paddles of 16 to 19 r.p.m. with one pulley setting and 20 to 60 r.p.m. with the other. With little experience, results can be translated to plant scale. Another factor in the use of this 'jar' test is that, even when results on the plant are satisfactory, tests carried out with the instrument will often lead to economical treatment without causing deterioration in effluent quality.

### Ultra-micro Sampling Accessories

Three new accessories developed for the Perkin-Elmer Model 2 spectrophotometer designed by Perkin-Elmer AG, Zurich, Switzerland (UK branch office, 160 Cheapside, London EC2), allow the



advantages of the pressed potassium bromide technique for the infra-red analysis of solids to be extended into the area of ultra-micro analysis.

The Perkin-Elmer 6X ultra-micro sampling system of condensing the infra-red sampling beam maintains high energy levels with analytical samples as small as 0.5 mm. in diameter.

Fabricated entirely of stainless steel, the Perkin-Elmer KBr Ultra-micro die consists of two separate parts for pressing KBr pellets of 0.5 mm. or 1.5 mm. diameter. The pellet size is controlled by an orifice of proper diameter in the centre of a 13 mm. stainless steel disc, in which the potassium bromide powder and sample are poured before pressing. The same vacuum which is used for evacuation is used to apply the pressing force. In making a sample disc of 1.5 mm. (.063 in.), a force of 500 lb. is required, supplied by a small arbor or hand screw press.

### Chemists Helped Design New Premier Mixer

The newly introduced Premier laboratory mixer, Model 1300, apart from conventional mixing and agitating, can be used for emulsifying, dispersing, wetting, pigmenting, reacting, dissolving and many other processes, state *Premier Colloid Mills Ltd.*, Brettenham House, Lancaster Place, London WC2. A unique feature is said to be the unit's high-speed turbine type head. Fitted with a 1/30 h.p. motor, the mixer has a maximum speed of 4,000 r.p.m. Speed control is positioned facing the operator and an on-off switch is integrally built in. The mixer is finished in grey stove enamel.

Priced £25, the model was designed after a survey of UK chemists to discover what features were sought in a laboratory mixer. A telescopic unit gives infinite variations in horizontal and vertical movements and eliminates the need for a full stand. A Dispersator mixing head is supplied for general use, but a marine-type propeller can be fitted for gentle agitation, or a small cage type beater can be fitted for thicker products.

### Fire Protection in the Laboratory

Two new items of fire extinguishing equipment recently introduced by the *Pyrene Co. Ltd.*, Brentford, Middlesex, are particularly suitable for laboratory fire protection. The constant pressure liquid extinguisher is a pressurised appliance that is clean, safe and rapid in action. It can be charged with either Pyrene fire extinguisher liquid, or chlorobromethane. Both of these form a heavy, dry, cohering blanket of vapour on coming into contact with flames, smothering fire instantly and completely. This extinguisher, which is nitrogen-pressurised, has a continuous discharge of 22 seconds. Its 'squeeze-grip' valve is so designed that the powerful jet can be released or stopped at will.

The dry chemical extinguisher, the smallest of a range of fast extinguishers for 'knocking out' oil or spirit fires, weighs less than 10 lb., complete with its charge of 5 lb. of Pyrene dry chemical

powder, and this lightness, coupled with its 'pistol-grip' operating head, allows the extinguisher to be operated using only one hand.

Powder is discharged through twin apertures in the nozzle, and is diffused into a fine cloud with a range of 8 ft. Discharge is complete in 8 seconds, but may be interrupted by releasing the trigger. When only partial discharge of the powder has been necessary, the residue of the charge can be saved for future use.

### Computing Counting Controller

The new type SA.501 computing counter controller, introduced by *Racal Engineering Ltd.*, Western Road, Bracknell, Berks, is said to be the first transistorised counter with in-line read-out to be made in the UK. It provides a universal equipment for laboratory for the measurement in laboratory and plant of frequencies, time intervals, events, batch counting and control.

Insensitive to transistor characteristics and component tolerances, it has a fully variable digital time base for direct display in desired units. Decades use reliable scale-of-10 ring counters. Range is random pulses and up to 50 kc/s. Accuracy is of the order of  $\pm 1$  count  $\pm$  crystal stability. Time base is variable from 1 millisecond to 10 seconds in 1 millisecond steps.

### Combustion Furnace

Among the range of laboratory apparatus available from *Radiation Group Sales Ltd.*, Radiation House, 7 Stratford Place, London W1, is the No. 2 combustion furnace which facilitates organic analyses using tubes of up to 1 in. outside diameter. Being formed of interlocking sections it can be supplied in different lengths. Each section is 6 in. long and is heated by five bunsen burners fed from a common gas supply. With a firebrick body and tiles, it has brass burners and control taps.

### Terminal Assembly

The Spadelok terminal assembly developed for the company's use by *Radiovisor Parent Ltd.*, High Path, London SW19, has since been made available to laboratories and educational institutions where a considerable amount of building up of experimental units takes place. The Spadelok is a 'plug-in' terminal assembly in which the electronic unit is released without disconnecting outgoing leads.

The design gives all the advantages of a plug-in construction, while eliminating costly plugs and sockets and terminal boards. Long leakage path makes the assembly ideal for low current signals. Maximum voltage is 440 volts; maximum current, 10 amps.

### Falling Ball Viscometer

The Koch multi-point falling ball precision viscometer, obtainable from *M. G. Reade*, Prospect House, Heath Street, London NW3 is a simple absolute instrument originally developed by Mr. Reade for his own use. Largely made of stainless steel, it can be used to test any fluid

or paste, whether transparent or opaque, in which a stainless steel ball will sink at a steady rate between about 4 and 40 mm. per second.

A simple test takes one minute, while full evaluation of apparent viscosity, plastic viscosity and yield value takes 5-10 minutes. Results are given in absolute units and are reproducible within 1 per cent. Completely portable and not dependent on electric supply, the range of the viscometer with apparent viscosities is from about 120 poises to about 900 poises, measured at a standard rate of shear of 0.5 seconds<sup>-1</sup>. Shear rate is from about 0.2 to 2.5 seconds<sup>-1</sup>; plastics viscosities from about 5 to 250 poises.

### Dynacap pH Meter

The use of a dynamic capacitor in the input stage of the new Pye Dynacap pH meter is said to provide exceptional zero stability, linearity and accuracy. Full advantage of this circuit is taken by the provision of expanded scales.

The basic scaling of the meter gives two main ranges of 0-10 and 4-14 pH units on a 5 in. scale, but the performance of the amplifier enables this to be expanded seven times so that the scale is, in effect, 35 in. long, allowing excellent discrimination. A lower scale reads from 0-2.0 pH units, each division being 0.02 pH. The range switch will select, besides the main ranges of 0-10 or 4-14 pH, sub-ranges of 2 pH units each—0.12, 2-4 4-6, 6-8, 8-10, 10-12 pH and 12-14 pH. This facility permits the most precise work to be carried out easily and speedily.

The circuit is essentially that of a sensitive valve voltmeter preceded by a dynamic capacitor input stage with a high



Dynacap by W. G. Pye and Co.

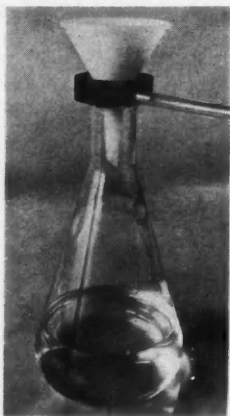
degree of overall negative feedback. The negative feedback gives almost perfect linearity, and normal mains voltage variations give practically no change in zero or in sensitivity. The absence of grid current eliminates errors normally encountered when using robust electrodes.

Both fully automatic and manual temperature compensation are incorporated. Parallel with the pH ranges are the main millivolt ranges 100mV and 400 to 1,400 mV, and expanded ranges of 200 mV throughout the full range 0-1,400 mV. Zero stability is such that total drift is normally less than  $\pm 0.03$  pH over 24 hour period.

This instrument was shown by *W. G. Pye and Co. Ltd.*, Newmarket Road, Cambridge, at the microchemistry symposium.

### Rediweld Filter-hold

Recently introduced by *Rediweld Ltd.*, Crawley, Sussex, is their Rediweld Filter-hold. This consists of a flat, soft rubber cylinder which provides a novel and efficient means of vacuum filtration with Büchner funnels. It obviates the need for tubulated filtering flasks in sizes of 250 ml. or greater and eliminates the necessity to prepare stoppers with con-



Filter-hold by Rediweld

necting tubes when Büchner funnels are to be used with round-bottom flasks or with bottles.

When using the filter-hold, the stem of the funnel is said to extend well below the vacuum connection so that filtrates cannot be carried over into the vacuum line. A 90° conical aperture holds the funnel in a position of maximum stability and the flat underside provides the necessary vacuum seal. Connection of the receiver to the vacuum source is made through the Filter-hold so that the latter, once connected with the vacuum line, is ready for use with most Büchner funnels on any receiver which can withstand evacuation and which has a neck or opening of 1½ in. to 3 in. diameter. Price is 18s. each.

### Photoelectric Anemometer

New instruments from the range of *Short and Mason Ltd.*, 280 Wood Street, London E17, include a low-speed photoelectric anemometer, a portable model, remote indicating low range electronic hygrometer, a 100,000-ft. barometer, Hirst pattern surface wetness recorder, and a Hygrosil relative humidity indicator.

The low speed photoelectric anemometer comprises two parts, measuring tube and counter unit, connected by an eight-way electrical lead. The measuring side consists of an aluminium tube 30 in. overall length with an extremely light vane, supported on a single jewelled pivot, mounted 20 in. from the inlet of the tube. The vane is made from .006 in. electron sheet and has eight blades, two (adjacent) of which are made from clear mica. The opaque blades

interrupt a beam of light focused from a low voltage lamp on to a photo-cell. The ratio of on to off time is therefore 1:3 and the blades are set at an angle of 45° to the air stream. A total count of 99,999,999 is available.

This company is about to release details of its Permafuse method of marking etched glass thermometers. The marking, being fused into the glass is not affected by strong chemicals and will withstand immersion in concentrated acids. Also being developed is a thermometer reader that enables the thermometer to be read more readily.

### Constant Weight Feeder

Latest models of the constant weight feeder, made by the *Sinex Engineering Co. Ltd.*, Central Way, North Feltham Trading Estate, Feltham, Middx, have been developed for such low outputs as 5 lb. an hour, coupled with extremely high accuracy. A vibratory feeder delivers the material from a storage hopper on to a short belt conveyor driven at a constant speed.

Electronic detectors are mounted on either side of the zero position of the scalehead and if the rate of flow from the feeder varies, the input current to the feeder is varied proportionately. Another detector transmits impulses of a strength proportional to the distance the indicator moves from zero in either direction and thus inhibits 'hunting'. It is claimed that pre-set output will be maintained to an accuracy of  $\pm 0.5$  per cent.

### Interchangeable Keys for Stopcocks

After much research into methods of manufacture and testing, *G. Springham and Co.*, Harlow New Town, Essex, have introduced stopcocks fitted with interchangeable keys. Specialists in this field, the company states that by using a new technique of grinding it is now able to produce both barrels and keys independently ground to very fine limits.

A special testing equipment developed by the firm ensures that each barrel and stopcock key is such that leakage rate is well below the British Standard specification. Only one size, 2 mm. bore, is at present being marketed, but a 4 mm. model will be put into production in the near future. The 2 mm. size is available in Pyrex as a stopcock, or fitted to burettes sizes 10 ml. to 100 ml. and incorporated in 50 and 100 cc. separating funnels. A catalogue is available on request.

### High Speed Ball Mills

Recent tests have shown that the high speed ball mill, Mk 1, developed by *Steele and Cowlshaw Ltd.*, Cooper Street, Hanley, Stoke-on-Trent, primarily for the paint industry, is even more successful when used on chemical, pharmaceutical and metal powders. Typical dry processing times claimed include tungsten carbide, 3 hours; cobalt-nickel-aluminium, 15 minutes; ferro silicon, 1

hour; pyrites, 1 hour; graphite, 8 hours.

The design incorporates four removable and interchangeable pots which are placed vertically and revolve at high speed around a central drive. The powerful centrifugal force set up holds the grinding media and charge against the wall of each pot. The pots are geared to revolve simultaneously in the opposite direction on their own axes, thus causing activation within the mass of the grinding media and charge. Pots can be changed over in 2 to 3 minutes.

Mk 1 is a small capacity mill for laboratory or production work and is powered by either a 1 or 3 h.p. motor depending on the specific gravity of the materials to be processed.

### Air Conditioning

Suitable for close control of temperature and humidity in laboratories, test rooms and standard rooms are the Air-monitor T1 and T2 models available from *Thermocontrol Installations Co. Ltd.*, 2-10 Valentine Place, Blackfriars Road, London SE1. In both models recirculated air quantity is up to 1,000 c.f.m.-28.3 m<sup>3</sup> min.; both fresh and exhaust air quantities are up to 200 c.f.m.-5.6 m<sup>3</sup> min.

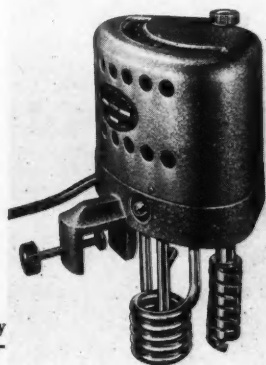
Total cooling of the T1 model is 12,000 B.Th.U. per hour—3,024 Kg/cal; dehumidification specification is 2,500 B.Th.U. per hour—630 Kg/cal; humidification 1.0 LW. Total cooling of the T2 model is 24,000 B.Th.U. per hour—6,048 Kg/cal; dehumidification is 5,000 B.Th.U. per hour—1,260 Kg/cal; while humidification is 2.0 KW.

Also available are Thermocontrol air conditioning cabinets which can accurately condition large rooms at a constant temperature  $\pm 1.0^\circ\text{F}$ . ( $0.6^\circ\text{C}$ .) and relative humidity to  $\pm 1$  per cent.

### Temperature Control Unit

Current design of the Tempunit temperature control device developed by *Techne (Cambridge) Ltd.*, Duxford, Cambridge, will maintain water bath temperatures up to 95°C within a limit of  $\pm 0.050^\circ\text{C}$ . An externally placed circulation device ensures a constant temperature throughout the bath (a litre of water per minute can be circulated).

Should the water level in the bath be lowered, due to evaporation, the Tempunit will, it is stated, automatically



Tempunit by Techne (Cambridge) Ltd.

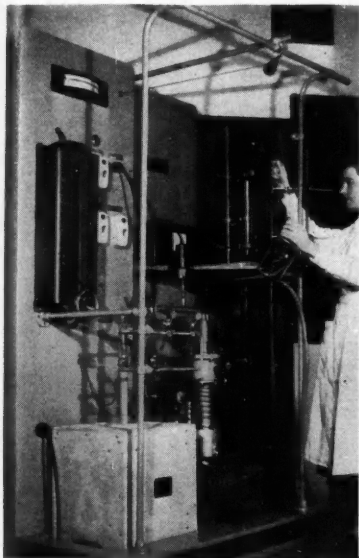
stop heating, thereby preventing damage to the bath and its contents.

This control instrument is easily attached to any vessel.

### 'Absolute' Filters

A range of 'Absolute' filters, Nos. 33, 44 and 55, are available from *Vokes Ltd.*, Henley Park, Guildford, Surrey, which are recommended for use in ventilating air entry supply and in preventing the introduction of cross contaminants from this source, when micro-chemical investigations are in progress. Although capable of extremely high dust retention efficiency, these filters have very low flow restriction characteristics.

### Determination of Hydrogen in Titanium



Latest metallurgical instrument of *Sutherland Thompson and Co. Ltd.*, Wells, Somerset, shown above, is for the determination of hydrogen in titanium by the hot extraction method

### Stereoscopic Microscopes

To meet the need for a simplified instrument without sacrifice of optical or mechanical performance, a new range of stereoscopic binocular microscopes has been produced by *W. Watson and Sons Ltd.*, Barnet, Herts. The basic instrument is supplied on a simple cast foot but can be readily fitted to a variety of different mountings for ease and convenience in various applications.

The image seen through the microscope is erect and three dimensional. The eyepieces, which may be either vertical or inclined at an angle of 45°, are adjustable to suit the interpupillary distance of the observer. The range of magnification is from  $\times 3$  to  $\times 140$ , with long working distances and large fields of view. The microscope can be supplied with a single nosepiece or with a rotating nosepiece to take either two or three

objectives. The objectives are parfocalled when fitted to the nosepiece.

Full details of this range of instruments are given in a special catalogue, List 6M.

### New Solidex Angle

Solidex unslotted steel angle is now available with a much improved stoved battleship grey finish at only a small increase in price, report the manufacturers, *Swifts of Scarborough Ltd.*, Olympia Works, Durham Street, Scarborough. In addition to the popular 1½ in. by 1½ in. size (16 gauge), 1 in. by 1 in. (18 gauge) angle is now being supplied; this is suitable for light laboratory stands, racks and fittings of all kinds, while the heavier material is recommended for stronger racks, trolleys, etc. The standard length for either size is 10 ft. but angle can be supplied cut to any length at a slight extra charge.

A hand punch is available to punch holes as required for bolting members together, and by using the Solidex marking-off system articles can quickly be put together.

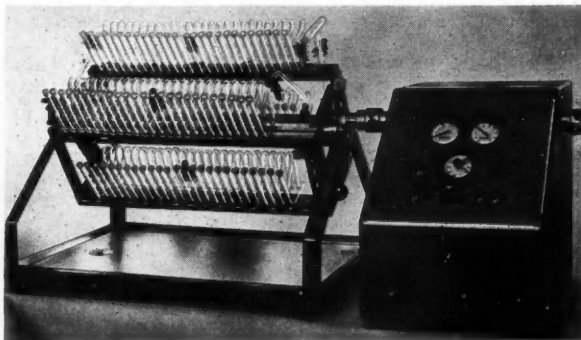
### Towers Apparatus for Micro-chemistry

For separations involving small quantities of materials from as little as 1 mg. upwards, *J. W. Towers and Co. Ltd.*, Victoria House, have a micro scale automatic countercurrent apparatus. The tubes with lower and upper phase volumes of 3 ml. each are specially designed to ensure efficient mixing, settling and negligible hold-up after transfer. They are fitted with polythene stoppers.

The tubes are robustly constructed in units of a convenient size for handling and these units together with an automatic upper phase dispenser are mounted in a supporting frame, the axle of which connects to a robot-drive mechanism. Frames are reading made up to carry as many micro countercurrent tubes as required. Each frame is equipped with a mechanical transfer counter. The 96-tube apparatus takes up a space approximately 3 ft. by 2 ft. excluding the robot. It can be manually operated.

For fractionating 0.5 to 5 ml. at atmospheric pressure or under vacuum, there is the Towers micro fractional distillation unit, which may be operated up to a temperature of 300°C.

Towers micro-scale automatic countercurrent apparatus



Principal features of this apparatus are: a vacuum jacket ensures that the column is operated under reasonably adiabatic conditions, thus achieving a high degree of separation; hold up in the column is reduced to a minimum by careful construction of the Vigreux column; the pear-shaped distilling flask permits distillation to a low bulk; the numbered and easily removed receiver and tubes facilitates the collection of fractions; the apparatus is made of Pyrex glass with standard ground joints throughout.

### Quart-size Laboratory Mixer by Winkworth Machinery

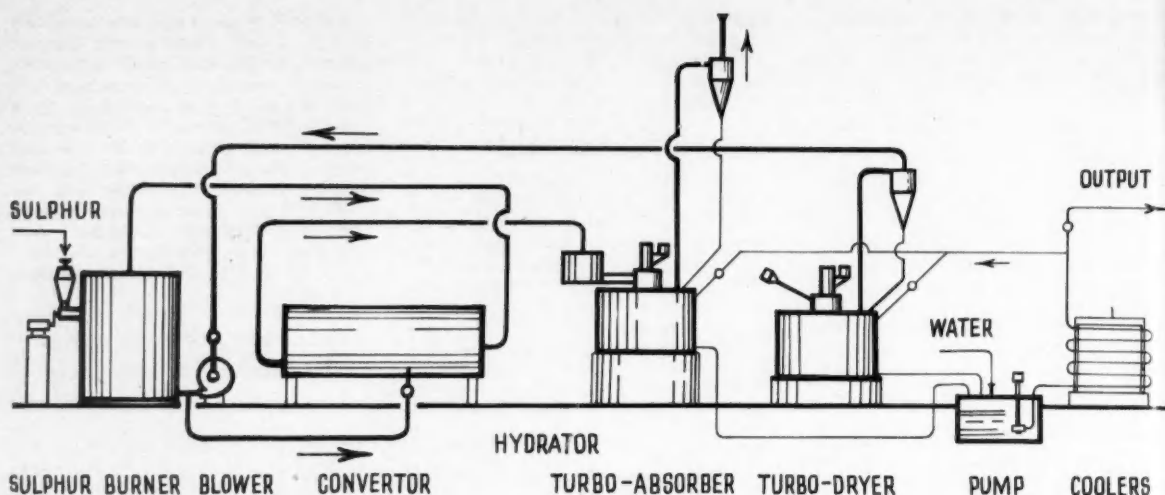
Several interesting points are claimed for the 1 quart 'Easy-to-Clean' laboratory mixer produced by *Winkworth Machinery Ltd.*, 65 High Street, Staines. This double 'Z' bladed mixer has an electrically heated trough for operation up to 250°C, inclusive of self-motorised drive (from a ½ h.p. geared motor, mounted on horizontal bed plate), and also an electronic temperature indicator control which can be preset for any given temperature from 0°C to 250°C.

The electrically heated pan can be easily and quickly interchanged either for a plain pan or a jacketed pan for steam heating. By purchasing a 1 pint adapter i.e. trough and blades, the mixer can be converted quickly to a 1 pint mixer. Alternative blade arrangements can be supplied such as Z blades with serrated edges, dispersion type blades, Naben type blades etc.; all are stated to be easily interchangeable in the mixer. Also at very little extra cost the mixer can be modified to work under vacuum—it is claimed that 29 vacuum inches are easily obtainable.

### Comparator Discs for Two Recent Chemical Tests

Comparator discs have been developed by *Tintometer Ltd.*, The Colour Laboratory, Waterloo Road, Salisbury, for two recent chemical tests. Free and combined residual chlorine in water can be determined using the diethyl-*p*-phenylene diamine (Palin DPD) method and the Lovibond comparator. The novel feature of this method lies in the use of compressed tablets which aside from being more convenient in use, permit of reagents being combined together to





## New Tab Moritz Sulphuric Process Uses Wet Catalysis

TO meet the need for small or medium size sulphuric acid plants for users who wished to be independent of outside sources of supply, the French firm of Moritz have just introduced a contact Tab Moritz system using sulphur as a raw material. Full details are available from the UK associates, Moritz Chemical Engineering Co. Ltd., 204 Earls Court Road, London SW5.

The plant (see simplified flowsheet) has been designed for the low cost production of 12 tons of acid monohydrate per 24 hours. It comprises a Moritz rotary type sulphur burner which, fed by solid sulphur and a combustion chamber, gives production of gas with 12 per cent  $\text{SO}_2$  content. After cooling, this gas is fed to a three-phase converter with six stages of the air injection type to reach correct temperature for catalysis. The  $\text{SO}_3$  gas produced then passes into an intermediate vessel called a hydrator and finally to a Moritz turbo-absorber with an acid cooling system; a blower ensures circulation of gases throughout the plant.

The plant takes up little space and for

the production of 98 per cent acid, standard materials may be used for its construction. If required the plant can be adopted to produce solids for use at varying degrees  $-54^\circ$  Baume or 67 per cent  $\text{H}_2\text{SO}_4$ , but this would add to the cost as it entails the use of anti-corrosive materials in the construction.

A plant of this new design has been operating in France for some time. It is claimed to be easily controlled, only an hourly check being necessary on the weight of sulphur fed into the burner and on the volume of water introduced into the turbo-absorber to maintain the desired degree of acid. Simple adjustment of temperatures and back draught enable the plant to operate with minimum supervision.

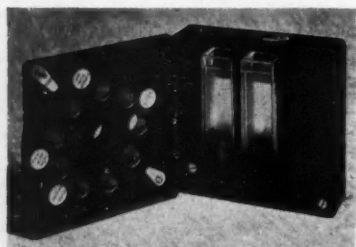
This type of plant can be supplied for outputs of up to 30 tons of  $\text{H}_2\text{SO}_4$  per 24 hours. It is also capable of the direct production of concentrated or diluted acid, or even oleum if necessary. Capital expenditure is claimed to be below that of a standard contact process plant with the cost of the acid accordingly reduced.

## Laboratory Equipment Review

(Continued from page 363)

give a procedure of exceptional simplicity.

For total residual chlorine, DPD tablet No. 4 (or Nos. 1 and 3) is used;



Tinometer comparator discs

for free and combined residual chlorine, Nos. 1 and 3; for free chlorine, monochloramine and dichloramine (also nitrogen trichloride), Nos. 1, 2 and 3.

The standard discs each contain nine colour standards; 3/40 A covers the range 0.1 to 1.0 p.p.m. chlorine; 3/40 B covers the range 0.2 to 4.0 p.p.m. chlorine. The discs require 13.5 m.m. cells or test tubes. A dulling screen, which is common to both discs, is required.

The chlorine values obtained by the use of these discs have been found to be identical with those obtained by the FAS titration of Palin. Dr. Palin has in fact tested and approved the master discs against which all reproductions are checked.

## UK's First Holmes-Kemp Nitrogen Generators

THE first two standard Holmes-Kemp nitrogen generators to be manufactured in the UK have been completed at the Turnbridge, Sheffield, works of W. C. Holmes Ltd. Each has a capacity of 2,000 c.f.h., but models giving ten times the output are projected.

The Holmes-Kemp plant generates nitrogen by the combustion of commercial fuel gases with air, and after the products have been cooled the carbon dioxide is removed by passing the gas through an absorber tower containing monoethanolamine.

The production system is completely self-contained and continuous, and the nitrogen produced can be stored under pressure.

## Building Lay-out Rephased At ICI's Terylene Plant

THE ANNOUNCEMENT that 700 out of nearly 2,000 men engaged on extensions to ICI's Terylene plant at Wilton, Yorks, are to be laid off before the end of the year does not imply any slowing-down of the expansion programme. The men are employed by outside contractors on the erection of buildings, some of which have now been found unnecessary following a rephasing of the plant layout.

Expenditure on development during 1958 will continue at the record level of £16 million established last year, and the long-term programme, it is stated, will continue with undiminished vigour.

## New Bradford Course

Details of three new courses are available from the Department of Chemical Technology, Institute of Technology, Bradford 7. These are: 'Techniques of polymer chemistry' (31 October and 1 November); 'Manufacture and dyeing of man-made fibres and plastics' (Mondays, 6 October to 8 December); and 'New metals and high temperature materials' (Wednesdays, 15 October to 10 December).

## Overseas News

### NEW AMMONIUM NITRATE PROCESS CUTS PRILLING TOWER BY THREE-QUARTERS

A NEW ammonium nitrate plant that will make prilled ammonium nitrate and a 60-40 prilled ammonium nitrate-limestone mixture with a prilling tower a fourth as high as those now used is being installed by the Ketona Chemical, Ketona, Alabama. The process has been developed by the Chemical and Industrial Corporation. The normal 200 ft. prilling tower has been reduced to a 50 ft. structure and the actual distance that the ammonium nitrate will fall inside the cooling chamber will be 20 ft.

The process makes it possible (*Chem. & Eng. News*, 25 August, p. 50) to use a smaller tower by first using a falling film evaporator to concentrate ammonium nitrate liquor so that it is almost free of water before being sprayed into the prilling section. Ammonium nitrate is converted to an 'anhydrous melt' by evaporation to 99.5 + per cent concentration. Pulverised limestone 100 per cent - 60 mesh, is mixed with the melt in steam heated tanks, usually in a ratio of 60 per cent nitrate to 40 per cent limestone. The melt is pumped to the top of the prilling tower, which is basically a spray chamber where ammonium nitrate falls countercurrent to an air stream.

The shorter prilling tower is said to make possible a 50 per cent or larger saving in construction costs, since the tower is the largest capital expense. Another advantage claimed is that prilled ammonium nitrate made by this anhydrous process is more dense and therefore more easily handled. The process is said to turn out less than 1 per cent fines. In addition a separate drying stage after prilling normally needed in plants that use a 95 per cent feed to the tower, is said to be unnecessary since the ammonium nitrate used is initially nearly anhydrous. This factor eliminates a stage which Chemical and Industrial feel to be the most troublesome part of the process.

#### Canadian Shell Step Up Petrochemicals Output

Shell's new detergent alkylate plant at Montreal East, the first in Canada to utilise a hydrofluoric acid catalyst, is in production with stocks for the soap industry. The catalyst, obtained from Valleyfield, PQ, is itself a relatively new product of Canada's fast-growing chemicals industry.

With the opening of its agricultural chemicals plant at Simcoe, Ontario, the Shell Oil Co. of Canada becomes the first Canadian oil company to devote an entire factory to the production of pesticides. These are being made from aldrin, dieldrin, endrin, phosdein and nemagon.

Shell are now planning to build a distillate hydrosulphuriser at its Shellburn refinery, North Burnaby, B.C. The contract has been placed with the Fluor Corporation of Canada Ltd., and completion is scheduled for February 1959. The trickle-phase hydrosulphurisation operation is a patented Shell process designed to reduce the sulphur content of distilled oils for heating, diesel and aircraft applications.

#### US-Unilever Talks on a Joint European Venture

The Cincinnati firm of Emery Industries Inc. and the Unilever group have opened talks on the possibility of the setting-up of a joint company in Western Europe for the production and sale of chemical by-products of the oil and fat industries.

#### Perchloryl Fluoride Oxidiser For Rocket Fuels

Pennsalt Chemicals, US, report that the US Government is investigating perchloryl fluoride as a potential oxidiser of liquid rocket fuels. Results are described as promising. It provides specific impulses with various fuels such as liquid fluorine, it is stable, easy to store and safer to handle than some other oxidisers.

Pennsalt are now supplying perchloryl fluoride in lots of up to 1 ton from a pilot plant, but output can be scaled up quickly, if required, the company report.

#### French Chemical Firms Form Algerienne de Pétrochimie

A new company formed to start a petrochemical industry in Algeria has been set up under the name of Compagnie Algerienne de Pétrochimie with an initial capital of £17 million. Subscribers are the French chemical firms of Ugine, Nobel-Bozel, Progil and Le Lebon et Cie, with the backing of two banks and a group formed to finance industrial development in Algeria. In due course the new company will use as a raw material the products of the Saharan oilfield.

#### Dow's Linear Polythene For Canada

Plans to produce linear polythene at their Sarnia, Ontario, plant have been announced by Dow Chemical of Canada. The company expects to begin production next year. No details as to size or cost of the plant have been made known.

For the past two years, Dow Chemical have been working on the development of modified linear polythene under the Ziegler low-pressure process. The new product is stated to be distinguishable from high-pressure polythene by its greater resistance to heat chemicals and vapour and its hardness, toughness and rigidity.

#### New Sulphuric Acid Plant Planned In Bolivia

A Bolivian firm, Fasyd, is planning to establish a plant near Viacha for the production of sulphuric acid. The necessary plant and equipment will be imported and the total investment involved will be \$150,000.

#### US Rubber Form Joint Italian Company

A joint company, Naugatuck-Rumianca, has been formed in Turin by United States Rubber and Rumianca of Italy. It will manufacture rubber accelerators and antioxidants, and various chemicals and specialities. Plans include the marketing of other Naugatuck Chemical (US Rubber's chemical division) products, such as plastics materials and synthetic rubber.

#### Two New Ghana Projects

Imperial Chemical Industries (Exports) Ltd. are to erect a factory at Tema, Ghana, to produce insecticides for sale to the Ministry of Agriculture. A salt specialist of the Israeli Salt Corporation of Tel-Aviv has arrived in Ghana to investigate the possibility of establishing a large-scale salt works.

#### International Congress On Natural Gas Processing

An International Congress on Hydrocarbons to be held at Piacenza, Italy, on 15, 16 and 17 September, will include papers on natural gas as a raw material for the chemical industry. M. Fouchier, of the Société National des Pétroles d'Aquitaine is to deal with the exploitation of the natural gas of Lacq, Dr. Buschmann, of Badische Anilin und Soda Fabrik will deal with the chemical processing of methane, while the utilisation of natural gas at the Vetrocoke factory will be discussed by a representative of the company. The Rumanian Ministry of Petroleum Industry is also to contribute a paper.

#### American Cyanamid Produce New Acrylonitrile Derivative

A new compound, sodium  $\beta$ -sulphopropionitrile, is now being made in pilot plant quantities by American Cyanamid Co., New York. Said to combine a number of unusual chemical properties the compound melts at a temperature of between 234°C and 244°C and is soluble in water, glacial acetic acid and hot methanol. Derived from acrylonitrile, it can, it is said, be used in the production of pharmaceuticals and surface active agents.

● **DR. JAMES MELVILLE**, director of the Waite Agricultural Research Institute at Adelaide University, has been appointed a part-time member of the executive of CSIRO, Melbourne, in place of Mr. H. J. GOODES, who has retired. Mr. Melville graduated in New Zealand, where he was born, and gained his M.Sc. with first class honours in chemistry. He was awarded a London Ph.D. degree after two years post-graduate work at the Imperial College, London. He was director of the plant chemistry laboratory of the New Zealand DSIR from 1939 to 1951, and appointed director of the grasslands division in 1955.

● **DR. ALAN J. HAYTER** has been appointed director in charge of the technical and sales division of Sharples



**Dr. A. J. Hayter**

Centrifuges Ltd., of Tower Works, Doman Road, Camberley, Surrey. He is transferring from an associated company, Sharples Chemical and Industrial Ltd., with whom he was concerned with the construction of nitrogenous fertiliser plants. Dr. Hayter graduated in chemical engineering at University College, London, and was awarded a Ph.D. for research work on high efficiency distillation columns with special reference to water isotope separation. Since 1954 Dr. Hayter has travelled extensively throughout the world in the application of continuous process methods.

● **DR. M. VAHRMAN**, senior lecturer in fuel technology at the college, has been appointed to a readership in industrial chemistry in the Department of Applied Chemistry, Northampton College of Advanced Technology, London, as from 1 September.

● **DR. P. A. LINTERN**, overseas controller, and Mr. S. G. TINSLEY, technical controller, have been appointed to the board of British Titan Products. Both have been with the company for over 20 years. Mr. N. D. HARRIS, former chairman of ICI (India) Ltd., has also become a director.

● **DR. J. D. NICHOLAS**, of the ARC Unit of Plant Nutrition (Micronutrients) Long Ashton Research Station, has been awarded the degree of D.Sc. by the University of London for his 'outstanding work in the field of plant biochemistry.'

● **DR. S. HUSAIN ZAHEER**, M.A. (OXON), D. PHIL.NAT. (HEIDELBERG), director of the Regional Research Laboratory, CSIR, Hyderabad 9, India, is planning to visit Europe for six weeks beginning 16 September. He will be contacting firms likely to be interested in the set-

## PEOPLE in the news

ting up of a 70,000 tons nitrogen fertiliser plant in the Andhra Pradesh coalfield. Dr. Zaheer's address on the Continent will be c/o Lurgi Gesellschaft für Warmetechnik m.b.H., Frankfurt Am Main, Lurgihaus, Gervinusstrasse 17/19, West Germany, while for his week's stay in the UK from about 10 October he may be contacted through Professor J. D. Bernal, F.R.S., Birkbeck College, University of London, Malet Street, London WC1.

● **DR. D. L. T. OPPE**, Mr. H. DEWEZ and Mr. C. I. BALL have been appointed directors of the Anglo-French Phosphate Co. Mr. F. J. PEDLAR and Mr. A. H. SMITH have resigned from the board. Control of 80 per cent of the company's capital is now held by Robert, Benson, Lonsdale and Co.

● **DR. M. B. GREEN**, B.Sc., Ph.D., A.R.C.S., A.R.I.C., who has been appointed chief research chemist with Calmic Ltd., manufacturers of vacuum shelf driers and other equipment, Crewe, was formerly a research chemist with May and Baker Ltd.

● **DR. M. J. RIEMERSMA** is from 1 September relinquishing full-time employment as Hercules Powder Co., Wilmington, Delaware, US. After having held

this position, with headquarters at The Hague, Holland, for more than 30 years. He will continue to act as part-time European consultant to Hercules.

● **MR. G. C. W. COMLEY** has left the NE Division of the Central Electricity Generating Board to take up an appointment as chemist with the Industrial Group, UK Atomic Energy Authority, at Winfrith, Dorset.

● **DR. J. S. MCPETRIE** has been appointed director-general of Electronics Research and Development, Ministry of Supply, in succession to Dr. D. H. BLACK.

● **DR. B. S. GIDVANI**, D.I.C., F.R.I.C., A.M.I.Chem.E., F.P.I., has been appointed to the board of Denton Edwards Paints Ltd., Abbey Road, Barking, as technical director.

### Will

MR. FRANK GEORGE ANDRAE, former sales director of Quickfit and Quartz, Ltd., Stone, Staffs, who died on 11 June last, aged 71 years, left £142,201.

### Changes in CA List of British Chemical Prices

THE FOLLOWING are price changes that have been recorded since 'British Chemical Prices' were last published in CHEMICAL AGE (26 July, p. 164):

**Potassium Iodide**. BP, under 1-cwt., per lb., 8s; per lb. for 1-cwt lots, 7s 3d.  
**Sodium Nitrate**. Chilean refined gran. over 98%, 6-ton-lots, d/d c.p., per ton £29.

**Tartaric Acid**. Per cwt.: 10 cwt. or more, £14 10s; 1 cwt. £14 15s.

**Dibutyl Phthalate**. In drums, 10 tons, d/d, per ton £210; 45-gal. drums, d/d, 1-4 drums, £216.

**Diethyl Phthalate**. In drums, 10 tons, per ton, £187 10s; 45-gal. drums, d/d, 1-4 drums, £193 10s.

**Dimethyl Phthalate**. In drums, 10 tons, per ton, d/d, £179; 45 gal. drums, d/d, per ton £185.

**Diocetyl Phthalate**. In drums 10 tons, d/d, per ton £284; 45-gal. drums, d/d, per ton, £290.

### New UK Company to Make Dowpon

A NEW agricultural chemical manufacturing company, Dow Agrochemicals Ltd., has been set up in the UK by the Dow Chemical Co., US, in partnership with Dr. W. E. Ripper, founder of Pest Control Ltd., now Fisons Pest Control Ltd. With head offices at 48 Charles Street, London W1, the new company will manufacture a range of Dow agricultural specialities in this country.

The first product on the development list for manufacture is the systemic grass weed-killer Dowpon which, it is claimed, can cheaply and easily solve the problems of farmers faced with the menace of couch grass. Dowpon is said to be fully herbicidal to couch and similar grasses and probably to bracken at a cost of £5 to £7 an acre. The product is being

imported until it becomes available from a new UK plant now in the planning stage. Negotiations on the question of a site are now being held.

Dowpon has been available overseas for some time and in the past few years has been extensively tested by various units of the Agricultural Research Council. Dowpon is the trade name for dalapon, 2,2-dichloropropionic acid. The present product is formulated as the sodium salt. Elliott and Fryer, of the ARC Unit of Experimental Agronomy (*Agriculture*, 1958, 65, 119-24) say this formulation is of particular interest because 'no other herbicide applied as a foliar spray is capable of killing grasses without affecting many broad-leaved plants'.



## Commercial News

### Albright and Wilson

As foreshadowed in the chairman's statement for 1957, the results of Albright and Wilson Ltd. for the first six months of 1958, although better than for the same period last year, are not as good as expected. Tax charges will be higher this year and present indications are that net profit for the full year will approximate to that of 1957.

Unaudited group results to 30 June show a trading profit of £2,453,000 (£2,234,000), before depreciation of £911,000 (£830,000). Profit before tax of £749,000 (£740,000) was £1,542,000 (£1,404,000). Group net profit for the period was £793,000 (£664,000), of which £47,000 (£18,000) was attributable to minority shareholders in subsidiaries.

An interim of 4 per cent on ordinary is to be paid for 1958 (equivalent to the 5 per cent interim paid last year before the recent one-for-four issue of fully paid shares).

### Boots Pure Drug Co.

Announcing that the interim dividend on ordinary shares is to be maintained at 3 per cent less tax, Boots Pure Drug Co. Ltd. emphasise that in view of the uncertainties of the present year this carries no implication in regard to the total for the year.

### Borax (Holdings) Ltd.

Statement from Borax (Holdings) Ltd. shows that trading profits for nine months ended 30 June were down to £1,171,770, compared with £2,639,534 for the corresponding period last year returning a net profit of £749,303 as against £1,850,458.

The new plants at Boron, California, have reached the rated daily production of the main products, and sales in the third quarter of the present financial year were regarded as satisfactory. While the tonnage of potash sold was higher during the third quarter than a year ago, prices were lower. A further reduction in potash prices for the new fertiliser year which began on 1 July will reduce earnings of the American operating company.

### ICI — Ilford Link

In an agreement announced last week on the development of the ICI colour photography process by Ilford Ltd., Imperial Chemical Industries are to acquire a £4.8 million one-third interest in Ilford's ordinary capital. ICI are to subscribe over a five-year period for 6.4 million new Ilford 5s ordinary shares at 15s each. Ilford's issued capital is currently in 190,000 £1 6 per cent cumulative preference shares and 13.6 million 5s ordinary shares.

The new Ilford capital is subject to the consent of the Capital Issues Committee which has been applied for. Ilford's shareholders will be asked to approve the necessary capital increase as soon as possible.

- **A. & W. Half Year Results Up on 1957**
- **£1.4 m. Drop in 9 Months Profits of Borax**
- **ICI—Ilford Link on New Colour Process**
- **Cut-back in Monsanto's Six Months Profits**

Under the new arrangement, Ilford in return for royalty payment will acquire the technical assets developed by ICI for colour photography and will undertake the manufacture and sale of colour photographic products. ICI will enter into a long-term agreement to undertake research into colour photography for Ilford. Ilford's colour process yields positive transparencies which can be viewed by projection or with a hand viewer; the ICI process introduced last year on a limited scale to professional photographers is a negative-positive system.

### Lawes Chemical Co.

Group net profit of the Lawes Chemical Co. for the year ended 30 June was £58,451 (£65,887) after tax of £70,368 (£74,633). Dividend on ordinary is 14 per cent (12½ per cent). The directors state that consolidated trading profit, although slightly down, still reflects a satisfactory trading year. Turnover was increased and the company more than maintained its share of the national increase in fertiliser sales. The reduction in profits is more than accounted for by higher spending on technical and advisory staff research, publicity and other expenditure.

### Monsanto Chemicals

Reduction both in net sales and in income in the first half of 1958 are reported by Monsanto Chemicals and its UK subsidiaries, but excluding those of the Australian subsidiary. Net sales have fallen from £7,930,927 to £7,489,849. (Net sales for the whole of 1957 were £15,683,272.) Before taxation net income totalled £531,742 against £870,738. After deduction of taxes estimated at £284,100 (£443,200) the net balance is £247,642 (£427,538). The 1957 balance was £864,374.

Interim dividend is being maintained at 5 per cent; total payment in 1957 was 13½ per cent.

Sir Miles Thomas, chairman, reports that the reduction both in net sales and in income is a reflex of the disturbed trading conditions that affected many UK industries during the first half of 1958. Adequate trading margins have proved difficult to achieve due to growing pressure of competition at home and particularly overseas. During the present period plants for new products have come into operation and manufacture of established products has been expanded, with consequent extra burden on the company. However, the benefits are now beginning to be reaped and should help the business during the second half of this year.

Towards the end of the half-year trading conditions improved, an experience that has since been maintained.

A meeting is to be called on 24 September to raise borrowing powers from the equivalent of the company's issued capital to the aggregate of the company's paid-up capital and group resources. Issued capital according to the last balance sheet was £8.4 million.

### Benn Brothers

Trading profit of Benn Brothers Ltd., proprietors of CHEMICAL AGE, for the year ended 30 June was £114,114 (£112,321). Profit before tax of £39,174 (£39,179) was £71,574 (£67,579). Final dividend of 10 per cent, making 15 per cent (same) has been declared on ordinary (C.A., 2 August, p. 199).

### Powell Duffryn

The Carbon Products Division of Powell Duffryn is actively engaged in development work involving the use of graphite in nuclear energy projects, and Professor J. M. Kay, head of the nuclear energy department of the Imperial College of Science and Technology, London, has been retained as consultant. Group trading profit for the year to 31 March 1958 was £2,675,815 (£2,789,922), and the ordinary dividend is maintained at 16 per cent.

### Chemische Werke Huls

While most of the plants of Chemische Werke Huls were fully employed last year, the production of butadiene on an acetylene basis was reduced since butadiene could be imported in the quantities needed and at favourable prices from petrochemical works in the UK and Italy. Acetylene supplies released were used to expand trade in solvents. Bottlenecks in acetylene and derivatives which still exist will probably be overcome by extending existing plants during the next few years.

This is stated in the annual report for 1957 of the directors of Chemische Werke Huls. At DM 523.1 million (£45.6 million), turnover rose by 9.6 per cent. Exports accounted for 35 per cent of the total. Net profit was DM 12 million (£1,042,000) and dividend payment was 10 per cent.

In a product review, the report states that sales of synthetic rubber dropped slightly with increased use of foreign cold rubber and the low level of world rubber prices. Under the heading 'detergent raw materials', the changeover in the company's working basis to tetrapropylene led to the expected rise in sales both inside and outside Germany.

Capital expenditure amounted to

DM 65.1 million (£5.5 million), of which DM 30.2 million (£2.58 million) was spent on manufacturing plants and DM 10.4 million (£880,000) on laboratories, technical centres and traffic installations. Included in the total was DM 16.1 million (£1.38 million) in respect of installations intended to supply Bunawerke Huls, in which Chemische Werke Huls hold a 50 per cent interest.

New installations included plant for the conversion of polystyrol; plant to produce tetrapropylene benzene as an intermediate for fully synthetic detergents; extension of styrol distillation plant; and a laboratory skyscraper.

### E I Du Pont de Nemours

Mr. L. du Pont Copeland, chairman of the finance committee of E. I. du Pont de Nemours, reports that sales are slowly edging upwards after having 'hit the bottom' of the US recession, and the graph for the rest of the year will be 'pretty horizontal.' Net income for the first half of 1958 was equal to \$3.08 per share, against \$4.3 last year. Du Pont will spend about \$240 million (£85½ million) on expansion this year.

### Du Pont of Canada

Sales of Du Pont Co. of Canada (1956) for the first six months of 1958 totalled \$40,241,000 (\$35,742,000). Net

income in the period was \$2,339,000 equal to 31 cents per share of common \$2,639,000 or 35 cents a common share).

It is stated that the effect of the increased volume of sales was offset by the high cost of operations in the early period of new plants which have yet to reach a profitable level of operations. Depreciation charges were up 33 per cent over the first half of 1957, because of the substantially larger investment in completed plants.

### Olin Mathieson

Sales and operating revenues of Olin Mathieson Chemical Corporation in the US and Canada in the second quarter of 1958 totalled \$148 million, an increase of 16 per cent over sales in the first quarter of this year and a decline of 4.3 per cent from sales of \$154 million in the second quarter of 1957.

Net profits were \$5,329,000, or 41 cents a share, an increase of 24 per cent over earnings of 33 cents a share in the first quarter. Profits were reduced by \$2,620,000, or 20 cents a share, by costs incurred for new operations in aluminium, high energy fuels and solid propellants and by idle plant costs of the Morgantown, W. Va., nitrogen plant. Second quarter profits in 1957 were \$9,705,000, or 73 cents a share.

### Changes in Export Licensing Control

FOLLOWING the relaxations in trade with the Soviet bloc and China (CHEMICAL AGE, 23 August, p. 293), the Board of Trade have made the Export of Goods (Control) (Amendment No. 3) Order 1958, which will come into effect on 1 September.

Under the Order control has been imposed on certain fluorinated silicone materials; certain liquid polymers; boron carbide; boron nitride; diethylenetriamine.

Control has been lifted from certain plastics materials and siloxanes; certain types of aluminium, alloys and powder; carbonyl iron powder; molybdenum carbides; vacuum pumps; synthetic rubber; balances; crystals of lithium or calcium fluoride; pH apparatus; spectrographic instruments and apparatus; barium nitrate; crude coal tar; cumene; dinitrotoluene; ethylbenzene; furfuryl alcohol; glycols and derivatives; hexamine; mono-ethylaniline; isopropyl ether; tetrahydrofurfuryl alcohol; titanium carbide.

The Order restricts the application of export control to a much narrower range of articles within the following description: metals and alloys; chemical and petroleum plant and equipment; compressors, blowers and fans; furnaces; microscopes; X-ray apparatus; molybdenum compounds.

### Chinese Visit Glassware Factories

An official delegation to promote trade with China recently visited the Staffordshire premises of Quickfit and Quartz Ltd., and Q.V.F. Ltd.

### Sunvic Open Process Control School

A TRAINING school to instruct customers' operators and maintenance staff in use of Sunvic pneumatic and electronic control instruments has recently been opened by Sunvic Controls Ltd., at their Harlow, Essex, factory.

Duration and content of the course is flexible, depending upon the degree of technical detail required by students, but it will not generally exceed one week. It will cover design, operation and applications of the pneumatic range of process control instruments, which are widely used in the chemical industry. In addition to lectures given by Sunvic technical staff, facilities are provided for practical instruction in a well-equipped laboratory which is a feature of the school. An interesting demonstration of Sunvic instruments under operating conditions can be given on a plant simulator and control panel.

The school which has for some time been providing instruction in use of Sunvic multi-channel pulse height analysers will be now administratively merged with the new process control school, although courses will normally be quite separate.

### Nylon Works Explosion

Imperial Chemical Industries have announced that their nylon works at Billingham-on-Tees, which were damaged by an explosion last week, will soon be back in full production. The explosion which took place while shifts were being changed caused considerable damage to the roof.

## Market Reports

### Steady Flow of New Business

**LONDON** A steady flow of new home trade business has been reported from most sections of the industrial chemicals market with the consuming industries taking good quantities against contracts. The movement to the textile trades remains slow and the demand for the agricultural chemicals is seasonably quiet. Shipment business continues to be fairly active and inquiries cover a wide range of chemicals. Phthalic plasticisers have been reduced in price otherwise the position shows little change and the undertone is firm. There has been little of fresh interest to report from the coal tar products market.

**GLASGOW** Considerably more activity was experienced during the past week in the Scottish heavy chemical market due to the fact that most industries have now resumed after the holidays. Demands both for spot and contract were maintained at a good level, together with placings for forward requirements. Prices generally remained unchanged and steady. There is still a fair demand for export coupled with a varied volume of inquiries.

**MANCHESTER** Prices for heavy chemical products have generally been maintained and actual changes of any consequence have been few. Demand for deliveries of the alkalis and other leading lines against contracts has been good, the position in this respect being healthier than during the height of the holiday season. A fair weight of new business has been reported for both home and export. In the fertiliser section, transactions are largely confined to those products in which early delivery rebates are available. Movement of coal-tar products continues steadily.

### Amendments to Soviet Bloc Embargo List

THE FOLLOWING amendments should be made to the list of goods embargoed to the Soviet bloc and China, extracts of which were published in CHEMICAL AGE last week, p. 293:

Under Group H (Metals, Minerals and Metal Manufactures): delete the item 'Cobalt (inc. scrap)' and insert: 'Cobalt and alloys containing by weight 50 per cent or more of cobalt (inc. scrap)'. Add to the items relating to nickel based alloys, niobium and alloys and titanium and alloys the words '(inc. scrap)'. After the item relating to minerals, raw and treated, insert 'Nickel powder.'

Under Group I (Chemicals, Plastics and Synthetic Rubber): In the reference to 'Silicone fluids and greases,' delete the item relating to lubricating greases and insert: 'Lubricating greases capable of operating at temperatures of 180°C or higher and having a drop point of 220°C or higher.'

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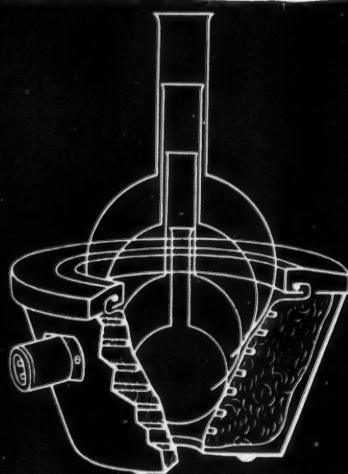
★ Elements easily exchanged.

★ And they cost less.

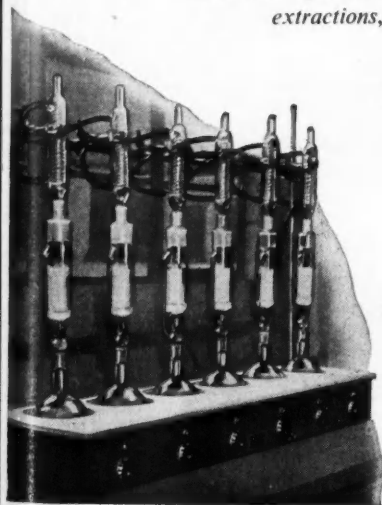
Isomantles are made for all flask sizes from the smallest micro mantle to glass plant of 200 litre capacity. Type "Multisize" (Patent No. 711365) allows heating of several flask sizes as shown in the diagram.

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*Catalogue LM describes Isomantles for Glass Plant, and new 44 page catalogue details Industrial Heating Mantles, Tapes, etc.*



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## Absorption spectrophotometry

By G. F. LOTHIAN

This book has been completely revised and reset for its second edition. It is concerned with the theory, instrumentation and application of absorption spectrophotometry, which is now one of the principal methods of industrial analysis and process control. There are few industries that do not employ spectrophotometers of some form or another in their laboratories. Mr Lothian, now a lecturer in physics at Exeter University, helped at one stage of his career to design several well-known instruments.

Price 52s. net

## The measurement of colour

By W. D. WRIGHT

This too is a completely revised and reset second edition. It is the standard work on a subject of vast industrial importance, a subject that concerns every manufacturer of coloured products. Professor Wright has a particularly lucid style, and no man could be better qualified to explain the intricacies of the fundamental trichromatic system. The book is fully illustrated with line diagrams and plates, many of them in colour.

Price 52s. net

## Clinical biochemical methods

By A. TARNOKY

The author, who is Biochemist at the Royal Berkshire Hospital, Reading, has arranged his book for the convenience of the worker on the bench in the pathological laboratory. The tests—all that are commonly encountered in clinical practice—are arranged in semi-tabular form for quick and easy reference.

Price 50s. net

## Spectrochemical Abstracts

Volume V of this well-known series, by E. H. van Someren and F. Lachman, is now ready. It abstracts all the important papers on spectrochemical analysis published in 1952 and 1953. Self-indexing under authors and subjects, it is the quickest and easiest possible source of information in a rapidly expanding subject.

Price 20s. net

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# TRADE NOTES

### Changes of Address

The Scientific Film Association has moved to larger offices in 3 Belgrave Square, London SW1 (Belgravia 6188).

The executive offices of Kelvin and Hughes Ltd. have been transferred to new premises at Empire Way, Wembley, Middx (Wembley 8888).

The Chemical Engineering Division of the APV Co. Ltd., which remained at Wandsworth, London, when the head offices and works of the company were moved to Crawley, Sussex, has now also been moved to Crawley. Its address is now Manor Royal, Crawley, Sussex.

### New Monsanto Bulletins

Monsanto Chemicals, 10-18 Victoria Street, London SW1, have issued four new technical service bulletins. One is from the plastics division describing Lustrex toughened polystyrene pipe (P 21/1), while the others are concerned with rubber processing; Flectol H antioxidant for rubber (12A/1), Monsanto chemicals for the footwear industry (13A/1), and pelleted accelerators for rubber (14A/1).

### Standard Heat-treatment Equipment

A policy of producing a range of standard equipments to cover the widest variety of heat-treatment applications has enabled Wild-Barfield Electric Furnaces Ltd., Otterspool Way, Watford By-Pass, Watford, to reduce delivery times. A number of furnaces ranging from laboratory muffles to medium size batch furnaces are available ex-stock. Delivery of many larger equipments and salt baths is said to be only a few weeks.

### Spray Dried Lecithin Powder

Moorgate Produce and Chemicals Ltd., 17-18 Basinghall Street, London EC2, have been appointed sole UK concessionaires for the spray dried lecithin powder of Hafen-Muhlen Werke. This new form of lecithin is milk based and has a pure lecithin content of about 30 per cent, equal to about 50 per cent commercial lecithin. The imperfect distribution of lecithin particles when the dry product is produced by mixing paste lecithin with wheat flour, etc., is said to be successfully overcome with the spray dried powder because of an excellent distribution of particles; when stirred with water a highly dispersive emulsion will be produced immediately, state Moorgate Produce and Chemicals. The new product can also be readily mixed with other powdered products.

### New BDH Catalogue Entries

New entries in the BDH catalogue are announced by the British Drug Houses Laboratory Chemicals Division, Poole. These are: arneil catalyst, a mixture of copper oxide containing 1 per cent ferric oxide; boron crystalline with an average particle size of 1-3 microns and containing 99.6 per cent boron (B) 2:6-di-tert-butyl-p-cresol (butylated hydroxy toluene); indane (hydrindene), an ingredient in turbo-jet engine fuels, supplied as a colourless liquid boiling at about 177°C

(d 20°/4° 0.962; n 20°/D 1.538);  $\alpha$ -methylstyrene (2-phenyl-propene), a pale yellow liquid, boiling at about 161°C (d 20°/4° 0.910; n 20°/D 1.539).

Added to the BDH range of amino acids is D- $\alpha$ -Alanine (laevo-rotatory)—CH<sub>3</sub>.CH(NH<sub>2</sub>).COOH. This new addition means that both stereoisomeric forms of the compound and the racemic mixture are now available. D- $\alpha$ -Alanine is a white powder and contains not less than 99 per cent C<sub>3</sub>H<sub>7</sub>O<sub>2</sub>N.

### SGB Protective Clothing

A comprehensive range of protective clothing is now being marketed by the newly-formed protective clothing division of Scaffolding (Great Britain) Ltd., Willow Lane, Mitcham, Surrey. Products include boiler suits, overalls, gloves, aprons, safety footwear, helmets, goggles, rubber boots, donkey jackets, waterproof clothing and duffle coats. Distribution of all these products is made from stocks held at the company's 36 branches throughout the country.

### Monsanto's New Catalyst

Monsanto Chemicals Ltd., 10-18 Victoria Street, London SW1, are now manufacturing in Britain Catalyst AC, a curing agent for amino textile resins. When used with urea formaldehyde, melamine formaldehyde or modified urea resins it promotes faster and more uniform curing and maximum fabric performance with minimum resin usage. Because of its stability and long bath-life, it is said to permit greater processing flexibility and to reduce the possibility of having to discard polymerised resin baths. It is compatible with most modifying and finishing agents, and is particularly effective with silicone water repellents, for which it is specifically recommended by manufacturers of waterproofing agents.

### Marinol Drying Oils

Chas. H. Windschuegl Ltd., Leadenhall Street, EC3, have been appointed UK agents for Marinol K and other processed marine oils produced by Marine Oil Refiners of Africa Ltd. Marinol K is a specially processed drying oil derived from fish oil through the Solxol segregation process.

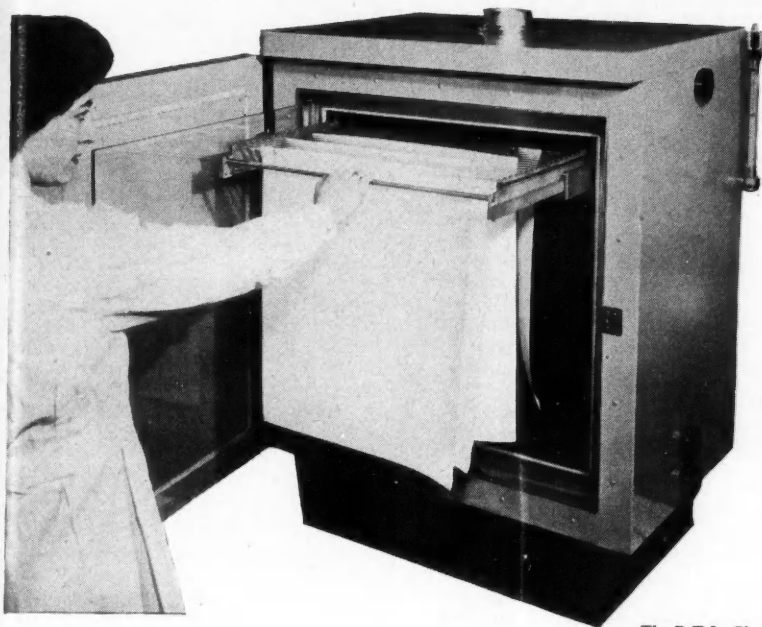
It can be used economically to replace linseed and other more expensive vegetable drying oils in a wide range of industries. Additionally the price of Marinol K is now considerably below that of linseed oil.

### New Protective Paint

Basic constituent of a new easily applied protective paint, developed by MacClester Chemical Co., 16, Mill Lane, Carshalton, Surrey, is zinc silicate. The paint is stated to protect metals for periods running into a number of years against atmospheric corrosion and corrosion caused by sea water, fresh water, electrolytic and galvanic action, and attack by carbon chemicals. The company claim also that the paint can resist very high temperatures.

# CHROMATOGRAPHY

## AND ELECTROPHORESIS

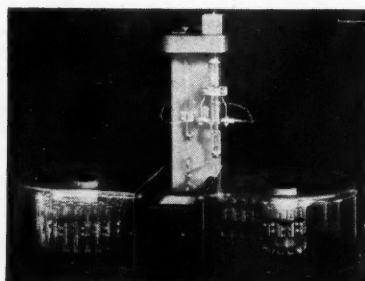
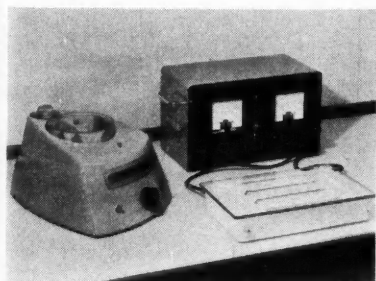


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## Company Meeting

# BENN BROTHERS LIMITED

THE sixty-second annual general meeting of Benn Brothers Limited, proprietors of *CHEMICAL AGE*, was held in Bouverie House, 154 Fleet Street, London EC4, on 29 August at 12.30 p.m. The following are extracts from the statement by the Chairman, Mr. Glanvill Benn, which has been circulated with the Report and Accounts for the year ended 30 June 1958:

Shareholders who follow the fortunes of Benn Brothers Limited from year to year will notice that the results now presented are very similar to those of a year ago. They reflect the sturdy strength and reliability of the Benn journals. Trade and industry need trustworthy information week by week. Violent changes in world raw material prices during the year, recessions in some markets, credit squeezes and relaxations, changes in interest rates, purchase tax alterations, hire-purchase restrictions, all these and similar alarms and excursions of the modern trading world, however unpleasant for those immediately concerned, serve to accentuate the need for prompt and accurate news and informed comment, as provided by your Company.

The shareholders should know that the all-important item of 'profit on trading' £114,114 against £112,321 a year ago, which appears to show no change worth remark, has, in fact, been achieved in spite of increased postal costs of £12,000, since the new GPO rates came into force last October.

I draw special attention to this item for three reasons—because it is beyond our control; because it forms an ironical comment on the practical effects of the Government's declared intention to en-

courage the export of British information; and thirdly because, self-evidently, revenues have again expanded to meet the increase.

Benn journals and books are read all over the world. We have paid subscribers in 115 countries, the nearest to the North Pole that I have been able to trace being in Rovaniemi, well within the Arctic Circle, and the most southerly in the Falkland Islands. We have always believed in personal contact with distributors of our journals, and also, as far as possible, with representative readers. First-hand knowledge of their requirements and the value they place on our services are of the greatest importance. During the early part of this year, Mr. Neil Wallace made a fact-finding tour of this kind visiting Brazil, Uruguay, Argentina, Chile, Peru, Ecuador, Colombia, Venezuela, Panama, the Central American Republics, Mexico, U.S.A. and Canada.

There are agents for the Benn journals and books in nearly all the countries of the world. I had the pleasure of renewing acquaintance with many of them, meeting others for the first time, and appointing new ones in countries not previously visited, during a flying journey round the world in the Spring.

One of the most widely known of our reference books, *The Newspaper Press Directory*, first published 112 years ago, is used in a number of countries—it would hardly be fair to particularize—as the only accurate guide to their own national Press.

During the year, another monthly serving the Spanish language markets, *Ingenieria Britanica*, was acquired, and merged with *Industria Britanica*.

Work has been in progress on an important addition to the Benn Group—the first post-war *Leather Trades' Year Book*—to appear in September. Published annually before the war by the United Tanners' Federation, this official reference book will now come from Bouverie House, sponsored by the Federation. There could be no clearer evidence of the standing and prestige of *The Leather Trades' Review*.

*The Electrical Journal* prestige series of articles on 'Diakoptics' by an internationally acclaimed authority has aroused much interest among mathematicians throughout the world, while the successful 'Electricity in the Oil Industry' series is to be published in book form at the request of the oil industry.

*The Red Book* is the most comprehensive record of electricity undertakings and keeps pace with the transition which is taking place in the supply industry, not only in Britain but throughout the world.

*Chemical Age* preview of trends in the U.K. chemical industry in 1958, the only one of its kind, was published in January. This journal has been in the forefront of reporting original scientific papers dealing with new developments in chemistry. All the major conferences and symposia have been covered, including those in Turin, Frankfurt and Brussels.

*Fire Protection Review* has broken new ground with an outstanding series of articles on the problems of fire fighting and prevention in the nuclear age.

Each year since the war, our income has grown. Over the period the increase has been of greater benefit to suppliers, the Postmaster-General, and inevitably, that giant non-playing shareholder, the Chancellor of the Exchequer, than to those who should be the beneficiaries, namely, the men and women who put up the capital to run this business, and the splendid team, many of whom I am glad to say are also shareholders, whose brains and energies produce the results now presented to you in this report and its accompanying accounts. I remain optimistic enough to hope that their continuing efforts, founded on knowledge and long experience, should ensure the maintenance of the company's revenues on the one side, and strict control of costs, which may even lead to a reduction, on the other.

I qualify my forecast by reminding shareholders that the Socialist Plan for Progress has been published. 'Go and find out what those children are doing; and stop them doing it' would not seem the frame of mind in which to run a great trading nation. Whether the Socialists have the chance to apply their schoolmaster methods is for the electorate to decide. If they do, a check to the expansion of British industry and to the expansion of Benn Brothers, Limited, may be expected.

## Chemical Engineering Practice

### Volume 4—Fluid State

General Editor: HERBERT W. CREMER, C.B.E., M.Sc., F.R.I.C.

Past President of the Institution of Chemical Engineers and Past President of the Royal Institute of Chemistry

The fourth volume in this comprehensive work on Chemical Engineering, is concerned with Thermodynamic Properties of Physical Systems, Transport Properties of Fluids and Measurement of Process Variables. *Chemical Engineering Practice* is published to serve as a link between Physics, Chemistry and Engineering to the better use of those sciences in the process industries. The work is available as a set of twelve volumes, or as individual volumes.

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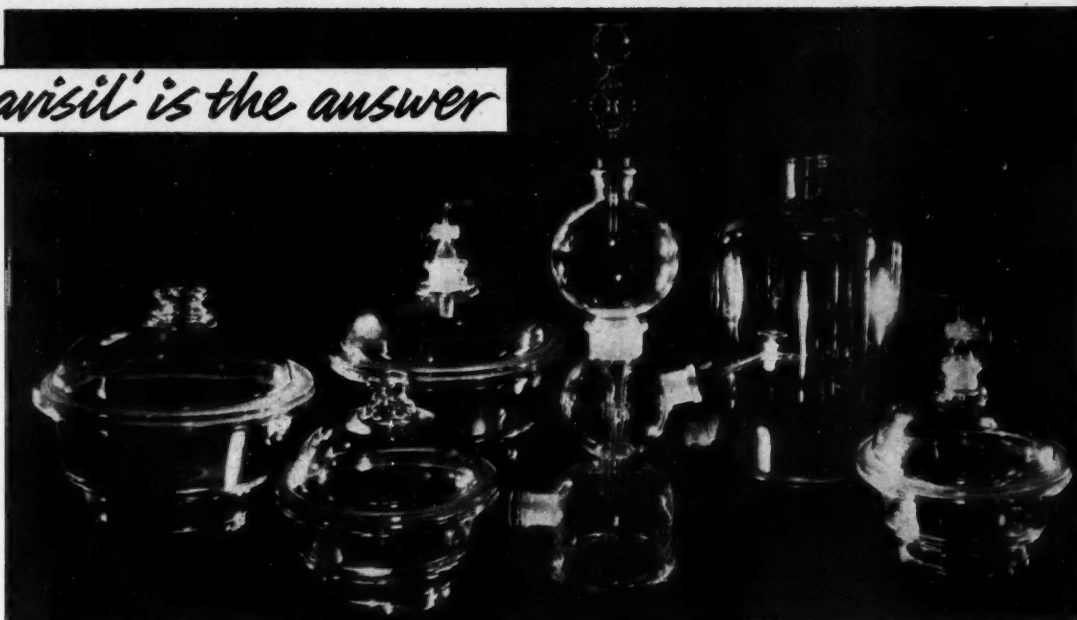
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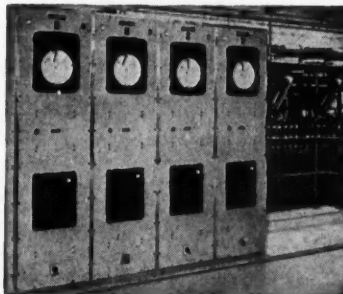
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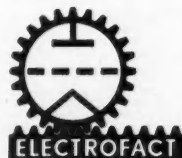
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# NEW PATENTS

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Specifications filed in connection with the acceptances in the following list will be open to public inspection on the dates shown. Opposition to the grant of a patent on any of the applications listed may be lodged by filing patents form 12 at any time within the prescribed period.

## AMENDED SPECIFICATIONS

On sale 10 October, or as soon as possible thereafter

Cellular polyvinyl chloride. Sponge Rubber Products Co. 716 696  
Benzene hexachloride. Stauffer Chemical Co. 777 290  
Inorganic oxide aerogels. Monsanto Chemicals Co. 784 392  
Therapeutic products of bacterial metabolism. Gratacos, M. L. 789 107

## ACCEPTANCES

Open to public inspection 8 October

Electrode elements. Vaughan, F. H. M., Gollop, H., Crennell, J. T., and Hill, F. L. 802 650  
Synthetic waxes, their manufacture and compositions containing them. Abril Corporation (Great Britain), Ltd. 802 727  
Electrolytic cells for the production of aluminium. British Aluminium Co. Ltd. [Jan. 14, 1954.] 802 471  
 $\alpha$ -Aluminium oxide. Egeysult Izzolampa Ex Villamossagi R.T. 802 731  
Condensers. Contraves AG. 802 680  
Electrodeposition of iron and iron alloys. Rockwell Spring & Axle Co. 802 503  
Dry-cleaning fibrous materials and preparations therefor. Farbwerke Hoechst AG. Vorm. Meister, Lucius & Brüning. 802 625  
Anthraquinone vat dyestuffs and process for their manufacture. Ciba Ltd. 802 681  
Production of naphthalene disulphonyl chlorides. General Tire & Rubber Co. 802 654  
Oriental films of substituted oxacyclobutane polymers. Hercules Powder Co. 802 683  
Conversion of gaseous olefins to motor fuel. Esso Research & Engineering Co. 802 734  
Betatrons. Brown, Boveri & Cie AG. 802 505  
Method of dip brazing aluminous metal. Aluminium Co. of America. 802 483  
Caffeic esters of quinic acid and quinate. Farmaceutici Italia, Soc. Anon. 802 668  
Antibiotic compositions for extending the storage life of foodstuffs. American Cyanamid Co. 802 736  
Process for the preparation of 1, 4-dicafeoyl-quinic acid. Farmaceutici Italia Soc. Anon. 802 669  
Organo-silicon compounds. Union Carbide Corp. 802 688  
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Copolymers containing an acrylamide. Soc. Nobel Francaise. 802 740  
Recovery of citric acid. Pfizer & Co., Inc., C. 802 522  
Thiobarbituric acid derivatives. Allen & Hanburys Ltd. 802 628

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Insecticidal compositions. Union Carbide Corp. 802 741  
Tertiary amines and their salts and process for the preparation thereof. Thomae G.m.b.H., Dr. K. 802 723  
Preparation of depolymerised levans. Farbenfabriken Bayer AG. 802 721  
Process for the production of yeast. Leopold, J., and Fencel, Z. 802 487  
Production of phenols and ketones. Distillers Co. Ltd. [Addition to 684 039.] 802 524  
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Polymerisation of acetylenic compounds. Imperial Chemical Industries Ltd. 802 510  
Polyester urethanes. Goodrich Co., B. F. 802 511  
Manufacture of spheroidal graphite cast iron. Regie Nationale Des Usines Renault. 802 440  
Polymerisation of olefines. Imperial Chemical Industries Ltd. [Cognate application 17935.] 802 633  
Tertiary amines and their salts and processes for the preparation thereof. Thomae G.m.b.H., Dr. K. [Addition to 802 723.] 802 724  
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Synthetic rubber-like materials. Imperial Chemical Industries Ltd. 802 531  
Densifying of magnesia. Armour Research Foundation of Illinois Institute of Technology. 802 515  
Manufacture of dihydroxyalkyl esters of terephthalic acid. Imperial Chemical Industries Ltd. 802 700  
Trypanocidal agents. Boots Pure Drug Co. Ltd. [Addition to 767 588.] 802 533  
Production of shaped bodies, coatings and impregnations from aqueous emulsions of copolymers. Farbenfabriken Bayer AG. 802 516  
Production of pseudo-ionones or homologues of pseudo-ionones. Givaudan & Cie Soc. Anon., L. 802 534  
Compounded hydrocarbon fuels. California Research Corp. 802 588, 802 589  
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Method of recovering uranium from ores containing it. Commissariat A L'Energie Atomique. 802 452  
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Treating fabrics with siloxanes. Midland Silicones Ltd. 802 540  
Alkylamine reaction products. Farbenfabriken Bayer AG. 802 541  
Filtering apparatus for industrial gases. Farrington Works & H. Pontifex & Sons Ltd., and Bainbridge, C. A. 802 764  
Preparation of non-corrosive hydrocarbon oils wholly or partly free from mercaptans and other sulphur compounds. De Bataafsche Petroleum Maatschappij N.V. 802 674  
Preparation of hypochlorite-treated hydrocarbon oils having reduced corrosivity. De Bataafsche Petroleum Maatschappij N.V. 802 675  
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